THE NICKEL BULLETIN

Monthly Abstracts of Recent Literature

JULY-AUGUST

1961 Volume 34 No. 7-8

THE INTERNATIONAL NICKEL COMPANY (MOND) LIMITED

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JULY–AUGUST 1961 VOLUME 34 No. 7-8

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

NICKEL

Surface Tension of Nickel: Effect of Crystallographic Orientation

H. MYKURA: 'The Variation of the Surface Tension of Nickel with Crystallographic Orientation.'

Acta Metallurgica, 1961, vol. 9, June, pp. 570–6.

The author gives details of a method for determining the orientation dependence of the surface tension of face-centred-cubic metals, in which interference microscopy is used to measure the grooves formed, during heat-treatment, at the intersection of twin boundaries with the surface. The orientation derivatives of the surface tension are obtained from the interferograms, and graphical integration of the curves of these derivatives plotted against orientation then gives the orientation dependence of the surface tension.

The application of the method to nickel specimens heated *in vacuo* is described.

Surface Energy and Surface Self Diffusion in Nickel: Effect of Impurity Adsorption

J. M. BLAKELY and H. MYKURA: 'The Effect of Impurity Adsorption on the Surface Energy and Surface Self Diffusion in Nickel.'

Acta Metallurgica, 1961, vol. 9, June, pp. 595–9.

The present paper was written to show that the variation of the surface free energy of nickel crystals with orientation (the γ-plot) is even more affected by impurity adsorption at lower temperatures than established in previous experiments at 1000°C., and that certain atomically rough planes may become planes of high surface mobility.

Striations were observed on nickel heated *in vacuo* at 900°C. The distribution of the striating orientations is regarded as suggesting the presence of point cusps in the plot of surface free energy at (100), (110) and (210) orientations and a line cusp between (410) and (510). There was evidence that surface diffusion is rapid for (110), and for a range of atomically rough planes between (410) and (210), in addition to the smooth (100) and (111) planes. Both effects are ascribed to impurity adsorption.

Diffusion of Nickel in Iron

K. HIRANO, M. COHEN and B. L. AVERBACH: 'Diffusion of Nickel into Iron.'

Acta Metallurgica, 1961, vol. 9, May, pp. 440–5.

Diffusion of nickel into iron was measured in the range 600°–1050°C. using radioactive Ni^{63} as the tracer element and employing the surface-decrease and residual-activity sectioning techniques.

Diffusivity below the Curie temperature was found to be lower than that anticipated from extrapolation of diffusion data for paramagnetic alpha-iron. The diffusion coefficients may, it is stated, be expressed as follows:

Gamma-iron, $D = 0{\cdot}77 \exp(-67{,}000/RT)$
Paramagnetic alpha-iron above 800°C., $D = 1{\cdot}3 \exp(-56{,}000/RT)$
Ferromagnetic alpha-iron below 680°C., $D = 1{\cdot}4 \exp(-58{,}700/RT)$

The anomolous decrease in the diffusion coefficient starting at about 800°C. (a temperature a little above the Curie temperature) is thought to be associated with the effect of short-range magnetic order on the formation energy of vacancies.

Diffusion of Niobium with Nickel

See abstract on p. 195.

Conference on Magnetism and Magnetic Materials

See abstract on p. 184.

Magnetization of Nickel under Hydrostatic Pressure

See abstract on p. 185.

Influence of Pressure on the e.m.f. of Nickel

See abstract on p. 192.

Critical pH and Critical Current Density for Passivity in Metals

See abstract on p. 194.

Rôle of Halide-Cycle Reactions in the Growth of Nickel Whiskers

R. E. CECH: 'The Rôle of Halide-Cycle Reactions in the Growth of Nickel Whiskers.'

Acta Metallurgica, 1961, vol. 9, May, pp. 459–63.

In recent years numerous contributions to the understanding of the solid state have been made possible by the availability of near-perfect metal crystals (whiskers). In this paper the author discusses findings relating to the growth of whiskers by hydrogen reduction of halide salts.

Study of the growth of nickel whiskers on nickel-oxide crystals, and of wüstite crystals on magnesium-oxide substrate crystals, revealed that nickel-oxide reduction in a near-equilibrium atmosphere, in the presence of halide and freshly reduced nickel, occurred by a halide-cycle mechanism. Investigation of the reduction of magnetite to wüstite in the presence of iron halide gave rise to a similar finding.

Evidence is presented to demonstrate that, in the case of nickel-whisker growth, an auxiliary reaction occurs at the base of the nickel crystals which could lead to non-uniform growth rates on different faces of the crystal and thus permit the Price-Vermilyea-Webb mechanism of whisker growth to operate.

Solid-Phase Reaction between NiO and NiS or Ni_3S_2

G. PANNETIER and J.-L. ABEGG: 'Solid-Phase Reaction between Nickel Oxide (NiO) and the Sulphides NiS or Ni_3S_2.'

Comptes Rendus, 1961, vol. 252, May 3, pp. 2724–6.

In the work reported the authors investigated the reactions occurring, under vacuum conditions, between (1) NiO and NiS and (2) NiO and Ni_3S_2. Variations in the mass of the solid phase were determined using a thermobalance, and the reaction products were identified by X-ray techniques.

Two distinct types of reaction were observed:

(1) The reaction between NiO and NiS progressed, at temperatures in the range 350°–600°C., as follows:

$$NiO+2NiS \rightarrow Ni_3S_2+\tfrac{1}{2}O_2.$$

(2) Ni_3S_2 and NiO reacted (but only at temperatures above 800°C.) to produce nickel:

$$4NiO+Ni_3S_2 \rightarrow 7Ni+2SO_2.$$

Under these conditions both reactions were total. Parallel to reaction (1), however, NiS decomposed at temperatures above 600°C. to Ni_3S_2 and sulphur and, in the region of 1000°C., Ni_3S_2 dissociated slowly to give nickel and sulphur.

Protection of Rubber Against Atmospheric Agents by Addition of Nickel Compounds

P. KARMITZ: 'A Contribution to the Study of the Protection of Rubber Against Atmospheric Agents. I. Examination of a Few Nickel Organic Compounds.'

Revue Générale du Caoutchouc, 1958, vol. 35, No. 10, pp. 1243–51.

A study was made of the suitability of 36 organic derivatives of nickel for use as stabilizers in natural rubber against sunlight and ozone attack. Nickel xanthates were found to be superior to both wax and commercially employed anti-ozonants under unstressed, statically stressed and dynamically stressed conditions. Use of such nickel derivatives produced a slight yellowish-black discoloration, but this defect could, it was found, be overcome by the addition of masking agents. It is concluded that nickel xanthates also act as accelerators in vulcanizing, and do not lead to degradation of the rubber at higher temperatures.

Ultra-violet absorption studies indicated that there is a relationship between the absorption spectra of the compounds and their protective action.

See also

M. BOUCHER and G. TESQUET: 'A Few Data on a New Protective Agent: the 'KPNi-1'.'

Revue Générale du Caoutchouc, 1958, vol. 35, No. 8–9, pp. 1077–83.

Following KARMITZ's findings (see previous abstract), the investigation described was undertaken to evaluate the protective action of nickel isopropyl-xanthate (available commercially as 'KPNi-1') in relation to different vulcanization systems.

Twenty-two basic compounds were prepared for examination. The compounds were made with various types of both natural and synthetic rubber, and for each elastomer the filler and vulcanization system were varied. The effects of adding different amounts of 'KPNi' were compared with those resulting from adding such preservatives as 'Ionol', 2246, 'UOP 88', 'Santoflex', AW, MTBZ or their mixtures.

It was apparent that 'KPNi-1' had no effect on the curing rate when the sulphur content of the mix was low. Addition of the nickel salt coloured the mix a pale-green shade, and no chemical transformation occurred during curing. When, however, the mix contained more than 2 per cent. sulphur, the vulcanization time was considerably shortened, and the mechanical properties of the vulcanizates were improved. From the grey colour conferred on the rubber, it is conjectured that the 'KPNi' is perhaps partially changed to nickel sulphide. The accelerating action of 'KPNi' was combined with an often much shorter scorching time, a defect which, it was found, could, however, be counteracted by the addition of a commercial retarder.

In oxygen-bomb ageing tests at 73°C. 'KPNi' showed a marked protective effect on all natural rubber—HAF black vulcanizates, but best results were obtained with mixtures of 'KPNi' and 4010. Both black and light synthetic rubbers offered satisfactory resistance

to thermal-oxidizing ageing when 'KPNi' was present, but, in this case, the protective effect of the salt was less than that of 'UOP 88'. Natural rubbers with a white filler were not protected by 'KPNi' alone.

The results of tests to determine the influence of 'KPNi' on rubbers exposed to strong sunlight indicate that, with the exception of phenolic anti-oxidants, the additive is a better anti-light agent than any of the other compounds studied. Vulcanizates containing quantities of inert fillers were not protected by 'KPNi'.

Under static-stress conditions, samples exposed to the atmosphere were best protected by wax, but 'KPNi' proved to be better than the other commercially available additives evaluated.

In dynamic open-air tests, using a machine of Mattia type operating at 5 cycles per minute, with 25 per cent. maximum elongation, 'KPNi' was found to confer on sulphur-vulcanized natural and GR-S compounds as high a resistance to cracking as 4010 or 'UOP 88'.

The authors draw the overall conclusion that 'KPNi' offers a wider range of protection than most available preservatives. 'In particular, it has a favourable action when there is a risk of network crackings, especially on light compounds. It is less efficient in this respect than phenols, but phenols do not protect at all against ozone cracking, while 'KPNi' is almost as efficient as paraphenyle-diamines, which, in turn, cannot be used with light compounds. Finally, it can exercise a protection against heat.'

Semi-micro Determination of Nickel with 4-Methylnioxime

R. E. LONGO: 'Semi-micro Determination of Nickel with 4-Methylnioxime.'

Microchemical Jnl., 1960, vol. 4, Dec., pp. 527-30.

After reviewing (with particular reference to the oximes) the factors to be considered in research concerned with the application of a new organic substance to inorganic analysis, the author discusses the application of 4-methylnioxime to the gravimetric determination of nickel on a semi-micro scale. The technique described is suitable for determination of nickel cation in amounts of 1-10 mg. in a volume of sample solution ranging from 25 to 150 ml.

Spectrophotometric Determination of Nickel, Copper, Cobalt, Iron and Manganese

G. H. AYRES and S. S. BAIRD: 'Spectrophotometric Determination of Copper, Nickel, Cobalt, Iron and Manganese as their Pyridine Thiocyanates: Simultaneous Determination in Mixtures.'

Talanta, 1961, vol. 7, Feb., pp. 237-47.

Since copper, nickel, cobalt, iron and manganese are often present together in various alloys, their simultaneous spectrophotometric determination offers obvious advantages. In a study of their determination based on the chloroform extraction of the metal pyridine thiocyanates, the authors found that optimum conditions require a pH of 5-8 and the presence of tartaric acid (to prevent precipitation of hydrous oxides). An excess of pyridine should be used, since chloroform extracts it readily from the aqueous solution. Improved results are obtained by making the extraction from a solution of high ionic strength (2 or above): the ionic strength can be varied by addition of magnesium nitrate. Perchlorate was found to decrease the absorbance, but the effect was essentially constant over a perchlorate concentration range of 0·8-2*M*.

An example is given of the simultaneous determination of copper, nickel, cobalt and iron in the same solution. Anions that also form metal pyridine compounds must be absent.

Gravimetric Determination of Nickel Using Respropiophenone Oxime as a Reagent

J. S. DAVE and A. R. PATEL: 'Respropiophenone Oxime as a Reagent for the Determination of Nickel.'

Jnl. Scientific and Industrial Research (*India*), 1961, vol. 20B, Feb., p. 81.

In this note the authors refer to results obtained in employing respropiophenone oxime as a reagent in the gravimetric determination of nickel (assuming the formula $Ni(C_9H_{10}NO_3)_2$ for the complex). Tests with nickel-chloride solutions containing from 7-30 mg. of nickel show that the metal can be determined with an accuracy in the region of $\pm$ 0·3 per cent.

Determination of Nickel and Cobalt in Soils

A. CAMUNAS, E. MINGARRO and A. BRU: 'Quantitative Determination of Elements in Minute Concentrations. I. Determination of Nickel and Cobalt in Soils.'

Anales de Fisica y Quimica, 1960, vol. 56 (A), May-June, pp. 121-4.

In the analytical method described, nickel and cobalt are extracted from soil by digestion with mineral acids, separated by paper chromatography (to avoid interference from iron), and determined spectrographically on platinum electrodes.

The technique is stated to be sensitive, rapid and economical, and therefore suitable for geochemical use. Sensitivity is in the region of 0·01γ/cc.

ELECTRODEPOSITION AND OTHER COATING METHODS

Symposium on Nickel/Chromium Plating

'Nickel-Chromium Plating.'

Published by *Robert Draper, Limited*, 1961; 140 pp. Price 25/-.

In November 1960 a One-Day Symposium on Recent Developments in Nickel/Chromium Plating was held

in London under the aegis of the London Branch of the Institute of Metal Finishing. This book contains the texts of the four papers presented at the Symposium and gives full details of the discussions to which each gave rise. The papers (see below) were abstracted, from the preprints then available, in *Nickel Bulletin*, 1961, vol. 34, No. 1, pp. 8-9.

H. C. CASTELL: 'Recent Developments in Nickel Plating', pp. 1-27; disc., pp. 56-76.

S. W. BAIER: 'Recent Developments in Decorative Chromium Plating', pp. 29-56; disc., pp., 56-76.

W. G. L. MILLER: 'Acceptance Requirements for Nickel-Chromium Plating', pp. 77-98; disc., pp. 114-32.

D. J. BOUCKLEY: 'Nickel-Chromium Plating to Specification Requirements', pp. 99-113; disc., pp. 114-32.

Brightening and Levelling Characteristics of Organic Addition Agents and their Effects on Stress in Nickel Deposits

T. SHIMUZU and K. AZADA: 'The Effects of Some Addition Agents in Bright Nickel Plating.'

Electroplating and Metal Finishing, 1961, vol. 14, Apr., pp. 126-8.

In the study described the authors investigated the brightening and levelling properties of certain organic addition agents, and determined the stress of nickel deposits produced from the plating solutions to which they were added.

Hull-cell tests were carried out to determine the brightening efficiency of pyridine, piperidine, nicotinic acid, propargyl alcohol, and compounds with triple bonds (such as 3-methylpentine-1-ol-3, butyne-1-ol-3 and 2-butyne-1,4-diol) in combination with saccharin and naphthalene sodium sulphonate. The results show that the mixtures containing triple bonds produced the most satisfactory brightening action. When the compounds were added in excess of 0·1 cc./L. of 35 per cent. aqueous solution, no deposit was obtained (except in the case of butyne diol) at low-current-density areas, and only a slight deposit was obtained at concentrations of 0·03 and 0·06 cc./L. Butyne diol had no brightening effect alone, but, in combination with 1 g./L. of saccharin, an addition of 0·2 g./L. yielded bright deposits, while 0·3 g./L. resulted in an excellent mirror finish. In beaker tests involving air agitation, addition of 0·08 g./L. of butyne diol with 1 g./L. of saccharin also produced bright deposits.

Levelling characteristics were evaluated using a Watts solution containing single or combined additions of saccharin, naphthalene sodium sulphonate and butyne diol. The copper substrates were scratched with 120-μ-deep grooves. The solution was operated at a pH of 4·2-4·5, a temperature of 45°C., and a current density of 1, 3 or 5 amp./dm.^{2}. Single additions resulted in no substantial improvement in levelling properties. In the case of combinations including butyne diol, on the other hand, the degree of levelling increased with increase in the butyne-diol concentration up to 0·2 g./L., and decreased with increase in current density and the sharpness and depth of the scratches. On the basis of these findings, the addition agents studied are classified into two groups, saccharin and naphthalene sodium sulphonate being primarily brighteners, and butyne diol a leveller.

The influence of organic additions on the stress of coatings deposited from Watts plating solutions was established using the Hoar method of measuring stress. Ethylene cyanhydrin, butyne diol and gelatin all produced tensile stresses, the intensity of which was directly proportional to the thickness of the electrodeposit and increased with increase in the concentration of the addition agent. Saccharin and naphthalene sulphonic acid (in concentrations of 0·2-5 g./L., respectively) produced compressive stresses. When a smaller quantity of saccharin was added, the compressive stress decreased, and, at a saccharin concentration of 0·05 g./L., a tensile stress resulted which was of the same order as that in deposits plated from solutions without any addition agent. Mixtures of the two groups of addition agent promoted a lower residual stress in the deposit.

Influence of Organic Additions on the Deposition Rate and Brightness of Electrolessly Deposited Nickel Coatings

W. MACHU and S. EL-GENDI: 'The Influence of Organic Materials on the Rate of Deposition and Brightness of Electrolessly Deposited Nickel Coatings. I.'

Werkstoffe u. Korrosion, 1961, vol. 12, Apr., pp. 223-30.

In earlier work the authors investigated the influence of the metal salts of cobalt, zinc, cadmium and uranium on the deposition rate and brightness of electrolessly deposited nickel coatings (*see* abstract in *Nickel Bulletin*, 1959, vol. 32, No. 7, pp. 212-3) and found that the heavy-metal salts differed in their effects. While cobalt salts increased the rate of deposition and were co-deposited with the nickel, the other metals, in particular cadmium, considerably decreased the deposition rate. Cobalt and uranium enhanced the brightness of the deposits up to about 40 per cent. of the reflectivity of a standard silver mirror; zinc produced greyish-black bright-nickel deposits with a brightness up to approximately 20 per cent., and in the presence of cadmium salts the coatings were matt, grey and of low adherence. At higher cadmium concentrations, nickel deposition ceased completely, and the nickel solution was found to be very sensitive to other metal salts at too high concentrations. Since organic as well as inorganic brighteners are employed in the electrodeposition of bright-nickel coatings, it was considered of interest to investigate the effects of organic brightening compounds when added to the sodium-hypophosphite electroless nickel-plating solution. The results of such an investigation (in particular, of the

brightness and deposition rate of the coatings produced) are reported in the present paper.

The plating solution, which was operated at a pH of 9·15 and a temperature of 82° or 92°C., was of the following composition:

	g./L.
Nickel chloride	30
Sodium hypophosphite	10
Sodium citrate	97·5
Ammonium hydroxide	47·5

Aluminium plate specimens were used as cathodes, and the brightness of the coatings was evaluated in percentages of the reflectivity of a silver mirror. The effects of the following organic addition agents were determined: alkaloids (e.g., cinchonine, caffeine and theobromine), sulphonamides (e.g., p-toluenesulphonamide), a sulphonomide (saccharin), nitrogen compounds (e.g., quinoline and pyridine), wetting agents, and formaldehyde. Data on the brightness, weight and thickness of the nickel coatings obtained are presented as a function of the concentration of the brightening agent. Photomicrographs typifying the structures of the coatings are included in the paper.

Both positive and negative catalytic effects were observed. Most of the organic materials investigated increased the brightness of the nickel, and the maxima, which in many cases occurred, did not correspond to the same concentration values as those associated with the maxima determined for the weight of the deposits. The most satisfactory brightness (corresponding to a completely smooth nickel surface) was obtained in the presence of p-toluenesulphonamide.

The surface structure of the nickel coatings was found to be governed by various factors. Whilst the organic brightening agents, in general, acted in a similar manner to inhibitors in reducing metal deposition in proportion to the concentration of the organic addition, optimum brightness occurred before the metal surface was completely covered with the brightener film, i.e., when a certain degree of porosity was still present. Only if the diaphragm permitted a constant rate of crystal growth, accompanied by a high rate of seed-crystal formation, both parallel and perpendicular to the metal surface, did the small homogeneous nickel crystals form which are prerequisite for formation of bright metal deposits.

Porosity in Electrodeposits

A. KUTZELNIGG: 'The Porosity of Electrodeposits: Causes, Classification and Assessment.'
Plating, 1961, vol. 48, Apr., pp. 382–7.

In view of the fact that porosity diminishes the protective value of most electrodeposited coatings, this paper was written 'to show the diverse forms that pores may take, to provide a classification system for these forms, and to suggest the causes that give rise to them'.

Pores in electrodeposits are defined as cavities, varying according to the method of formation of the coatings, and filled with air or other foreign matter. Cavities are subdivided into pores and cracks, which are further classified on the basis of size and according to whether they extend through the coating (transverse cavities) or not (masked cavities). Suggested classifications are tabulated and the types of cavity are illustrated.

Causes of porosity in electrodeposits are reviewed, the relationship between porosity and thickness of the deposit is considered, and, finally, reference is made to the principal methods employed for assessing total porosity and indicating local distribution of pores. Discussion of methods of revealing local distribution of pores (to which most attention is given) is confined to those applicable to nickel deposits. For detecting transverse pores in nickel coatings, the author suggests the use of the non-corrosive agent α-nitroso-β-naphthol, which produces green spots where the metal is exposed.

Electrochemical Reactivities of Various Nickel Electrodeposits

V. HOSPADARUK and J. V. PETROCELLI: 'The Relative Reactivity of Various Nickel Electrodeposits.'
Plating, 1961, vol. 48, May, pp. 479–87.

Commenting on the circumstances which led to the initiation of the work reported in this paper, the authors refer to the fact that, in studies of the corrosion-resistance of nickel/chromium electrodeposited coatings, neither the accelerated- nor the static-exposure tests now in use yield information which may be applied directly to the determination of the causes of failure or the mechanisms of corrosion, and that, although some electrochemical data have recently appeared (see, for example, abstract of paper by DUROSE in *Nickel Bulletin*, 1961, vol. 34, No. 3, pp. 58–9), a real need exists for basic data on the corrosion characteristics and behaviour of nickel and nickel/chromium electrodeposits. A comprehensive research programme designed to obtain pertinent electrochemical data is under way in the laboratories of The International Nickel Company, Inc., and the work reported by the authors was undertaken to determine the relative electrochemical reactivity and pitting tendency of various types of nickel electrodeposit. (Subsequent communications will deal with polarization and probable corrosion mechanisms.)

The foil specimens prepared for study were deposited from proprietary bright-nickel and conventional Watts-type plating solutions. In the case of those produced from the Watts bath, a study was made of the effect of pH and temperature on the electrochemical reactivity of the deposit. The influence of the chloride-ion concentration was established in work on foils obtained from Watts, sulphate/chloride, and all-chloride solutions, and, in addition, experiments were carried out on semi-bright and bright nickel foils prepared from Watts baths containing various addition agents. The solution compositions, plating conditions, and appearance of the deposits

obtained are noted in the paper. Specimens deposited in the presence of sodium thiosulphate or p-toluenesulphonamide (with and without coumarin), and all the commercial bright-nickel coatings, had a sulphur content of 0·03–0·01 wt. per cent., while the sulphur content of all the other deposits studied was much lower (0·002–0·004 per cent.). None of the foils was chromium-plated.

The electrochemical reactivities and relative pitting tendencies of the deposits were studied using potentiostatic and galvanostatic techniques (the suitability of these techniques for use as a basis for improved accelerated-corrosion tests is discussed). After testing, the 1·7-mil-thick nickel foils were examined microscopically and in the light box to determine surface pitting and perforations. Most of the test data were obtained using an air-saturated 3 per cent. (0·5 *N*) sodium-chloride solution at a pH of 6·0–6·5, but some experiments were carried out in air-saturated 3·6 per cent. (0·5 *N*) sodium-sulphate solutions, the pH of which was adjusted to 2, 4 and 6 by sulphuric acid. In one set of experiments the nickel was anodically polarized at a constant average current density of 2 microamp./cm.^{2} for 2½ hours, and the electrode potential was recorded against time, and the amount of surface pitting and number of perforations noted. In other experiments the nickel was anodically polarized for 2 hours at a constant potential 100 millivolts more noble than its steady-state open-circuit potential, and the average current density was recorded with time.

The data presented by the authors indicate that sulphur content has a major effect on the corrosion behaviour of the deposits. Where the sulphur content was 0·03 wt. per cent. or higher, the deposit exhibited a higher relative electrochemical reactivity and a greater tendency to pitting. This finding was true irrespective of whether the deposits were semi-bright or bright. There was also evidence that the presence of nickel sulphide in the nickel deposit could account for enhanced reactivity. The authors' studies suggested that small crystallite size and brightness do not greatly affect the corrosion behaviour of nickel deposits.

Since the reactivity of the nickel deposits plays an important rôle in the corrosion behaviour of decorative nickel/chromium coatings, the relative reactivities and potentials discussed in the paper are considered very pertinent, and nickel of greater reactivity should (all other conditions remaining constant) corrode more rapidly. The results indicate that, in corrosive media with a pH of about three and above, sulphur-containing deposits are characterized by a lower corrosion-resistance than sulphur-free. On the other hand, it is pointed out that the more-reactive nickel coating should sacrifically protect an underlying sulphur-free deposit (such as is found in some of the commercial double-layer-nickel coatings), and the data presented are regarded as confirming and explaining the superior results obtained with such double-layer-nickel systems.

The differences between the reactivities of the sulphur-containing and sulphur-free deposits tend to become less pronounced in chloride-free media at low pH, a tendency which appears to correlate with observations that the behaviour of the various deposits in marine environments is different from that in some industrial atmospheres where chlorides and pH are significantly lower.

It is noted that work still in progress suggests that the difference between the reactivity of sulphur-containing nickel and sulphur-free nickel involves the passivating tendency of the metal, in addition to an 'activation' type of reactivity. Sulphur-free deposits appear to passivate much more readily than the sulphur-containing coatings, and this factor may be influential in determining corrosion behaviour, particularly in the case of coatings without a final layer of chromium.

Microscopical Study of Nickel/Chromium Coatings after Atmospheric Corrosion

D. W. ERNST and F. OGBURN: 'The Nature, Cause and Effect of the Porosity in Electrodeposits. VII. A Microscopic Examination of Nickel-Chromium Coatings after Atmospheric Corrosion.'

Plating, 1961, vol. 48, May, pp. 491–7.

In much of the work carried out to study atmospheric corrosion of nickel-plated steel, the nature and extent of corrosion have been assessed solely on the basis of visual observations. For an understanding of the corrosion process, more information is, however, needed than is obtainable from visual appearance: data are, for example, required which will throw light on the microscopic nature of the corroded surface, on the history of the defects which ultimately lead to rust spots, and on the galvanic effects which are of significance. In this paper, the final report of work carried out within the scope of A.E.S. Research Project No. 13, the authors record the findings of work exploring some of the new approaches to the study of nickel-plated steel.

The paper is in three sections. The first is given up to a discussion of the results of microscopical examination of steel specimens which, plated with the following coatings, were exposed in a sea-coast and an industrial atmosphere: Watts nickel (unbuffed; buffed; buffed and then coated with chromium); bright nickel; bright nickel + chromium; double-layer nickel (semi-bright + bright); double-layer nickel + chromium. The nickel coating was about 38μ (1·5 mil) thick; chromium, where employed, was of the usual decorative type and about 3μ (0·01 mil) thick.

Consideration of the failures observed in this work posed the questions as to how they develop during the earlier stages of the corrosion processes, and what defects were originally present in the coating which would contribute to their occurrence. In the second phase of the programme four panels plated with bright nickel (two with chromium overcoats) were exposed to the same atmospheric environments as those in the first phase of the study. The panels were subjected to microscopical examination before exposure and at various intervals (ranging from three days to three weeks) during exposure, and the conclusions drawn from the data obtained are summarized in the second section of the paper.

Concurrently with these corrosion tests, the authors carried out an exploratory study of the behaviour of galvanic couples, such as might be encountered during the corrosion of nickel/chromium-plated steel. The data derived from study of nickel/steel, nickel/chromium and Watts-nickel/bright-nickel couples form the subject of comment in the third section of the paper.

Photomicrographs illustrating the types of corrosion suffered by the various coatings studied are included in the paper. It is shown that, in the presence of a chromium overcoating, bright nickel was perforated more by pitting corrosion than was Watts nickel, but that the latter coating contained many more micropits. Pits which did not extend to the basis metal were similar in appearance, irrespective of the type of nickel deposit or corrosive atmosphere involved. Cracks were found to occur in chromium-plated bright nickel after formation of pits in the nickel.

The authors' observations of the behaviour of the galvanic couples investigated revealed the polarity of the nickel/steel couple to change with variations in the electrolyte, and differences in the shape of the pits and in rust exudation associated with sea-coast and industrial atmospheres are considered explicable in terms of these changes. The polarity of nickel/chromium couples was found to change unpredictably in several electrolytes, and the chromium was usually found to be the anode. The nickel/bright-nickel cell was more active than any of the nickel/chromium cells.

Sulphamate Plating Solution for Deposition of Cobalt-Nickel Magnetic Coatings

I. TSU: 'A New Cobalt-Nickel Sulfamate Bath.'
Plating, 1961, vol. 48, Apr., pp. 379–81.

Thin cobalt-nickel films, which find wide application as magnetic-recording media, can be deposited from several types of solution, including those of Watts, hypophosphite and sulphamate type. In this paper the author gives particulars of a sulphamate solution (composition and operating conditions are reproduced below) which produces cobalt-nickel coatings having a wide range of magnetic properties and offers advantages over plating solutions at present in use.

Co^{++}	4·96 g./L.
Ni^{++}	14·85 g./L.
$NH_2SO_3^-$	59·40 g./L.
Cl^-	6·65 g./L.
pH (KOH)	6·5–6·8
Temperature ..	Room temperature
Typical plating voltage	1·7 volts
Cathode efficiency ..	100% (at 4·5 amp./sq. ft.: 0·486 amp/dm.²)

The solution may be made up using either cobalt chloride and nickel sulphamate or cobalt sulphamate and nickel chloride. Excess sulphamic acid is used to achieve a suitable concentration of $NH_2SO_3^-$.

Bath precipitation at the pH range quoted is not a problem, and Hull-cell measurements showed the solution to exhibit good throwing power. Bright deposits were obtained at current densities in the range 1–5 amp./sq. ft. (0·11–0·54 amp./dm.²). The coating deposited from this solution exhibited a coercive force of 500 Oe.

The composition of the deposits can be varied by varying the cobalt/nickel ratio of the solution. Boric acid, which is normally used in sulphamate solutions as a pH buffer, is not needed. The presence of chloride ions is regarded as important, since they increase the limiting current density at the cathode. Too high a concentration of sulphamate ions adversely affects the activity of the chloride ions.

NON-FERROUS ALLOYS

Pressing and Sintering of Nickel Silvers

J. WILCOCK and R. J. WOOLFALL: 'Pressing and Sintering of Nickel Brasses.'
Metallurgia, 1961, vol. 63, Apr., pp. 167–73.

Sintered nickel-brass components (in view of the fact that nickel silvers form homogeneous solid solutions comparable to alpha brass, the authors deem it more appropriate to refer to them in this paper as nickel brasses) are fabricated under conditions similar to those used for standard brass sintered components. In applications requiring a decorative appearance and/or corrosion-resistance, sintered nickel brasses offer advantages over the latter materials, however, and are used for various small parts in the automobile and aircraft industries, and in domestic food equipment. An interesting potential application for sintered nickel brass is as a material for keys for tumbler locks, an application which demands adequate corrosion-resistance, good torsional strength and maximum resistance to deflection. Alloys containing iron and phosphorus have been developed to meet these requirements. Since design requirements often render final machining operations necessary, and since the addition of small quantities of lead is standard practice to provide free-cutting properties, within the scope of the study now reported (which was carried out to investigate the pressing and sintering behaviour of a series of 10 per cent. nickel brasses) a study was made of the effect of an alloying addition of 2 per cent. lead. Standard sintered brasses were included in the test programme to provide a basis for comparison.

The data obtained are discussed in two sections concerned with the effect of the following on the mechanical properties of a 60-10-30 copper-nickel-zinc alloy: (1) powder characteristics and compacting and sintering conditions; (2) copper content and alloying additions of lead, iron and phosphorus.

For optimum results it was found necessary to lubricate the nickel-brass components by means of lithium-stearate additions and to compact at pressures in the range 30–35 t.s.i. (47–55 kg./mm.²: 67,000–78,500 p.s.i.). A reduction in copper content, and

Mechanical Properties of Nickel Brasses, Leaded Brass and 70–30 Brass
(Compacting Pressure 30 tons/sq.in.)

(*See abstract on p.* 182)

Material	Sintering Temperature °C.	Solidity %	Ultimate Tensile Stress	Elongation %	Hardness (B.H.N.)
80–20 Leaded Brass	880	90	28,000 p.s.i. (12·5 t.s.i.: 19·5 kg./mm.^{2})	20	48
70–30 Brass	880	88	28,000 p.s.i. (12·5 t.s.i.: 19·5 kg./mm.^{2})	8	50
70–10–20 Nickel Brass	940	90	36,000 p.s.i. (16·0 t.s.i.: 25 kg./mm.^{2})	16	57
60–10–30 Nickel Brass	880	88	33,000 p.s.i. (14·5 t.s.i.: 23 kg./mm.^{2})	10	65
60–10–30 Nickel Brass +1·5% iron	880	89	35,000 p.s.i. (15·5 t.s.i.: 24·5 kg./mm.^{2})	12	68
60–10–30 Nickel Brass +1·5% iron + 0·5% phosphorus	850	86	36,000 p.s.i. (16·0 t.s.i.: 25 kg./mm.^{2})	8	75
60–10–30 Nickel Brass +2% lead	880	89	26,000 p.s.i. (11·5 t.s.i.: 18 kg./mm.^{2})	8	65
50–10–40 Nickel Brass	880	80	32,000 p.s.i. (14·5 t.s.i.: 22·5 kg./mm.^{2})	3	73

addition of phosphorus, permitted the use of lower sintering temperatures, and replacement of part of the copper content by nickel improved the strength, ductility and hardness of components produced under the same conditions. Mechanical properties were improved still further by addition of 1·5 per cent. iron. Controlled additions of iron and phosphorus resulted in torsional properties approaching those of rolled nickel-brass strip.

The mechanical properties of the sintered brasses studied are compared in a table reproduced above.

Sulphamate Plating Solution for Deposition of Cobalt-Nickel Magnetic Coatings

See abstract on p. 182.

Oxidation Behaviour of Nickel-Aluminium Alloys

See abstract on p. 192.

Properties of Lead-Cadmium-Nickel Alloys

D. N. WILLIAMS, K. R. GRUBE and R. I. JAFFEE: 'Wrought Lead-Cadmium-Nickel Alloys.'

Trans. Amer. Soc. Metals, 1961, vol. 54, Mar., pp. 96–105.

Most lead-base alloys recrystallize at, or only slightly above, room temperature, and are generally characterized by poor resistance to creep at this temperature. The investigation reported was undertaken on the assumption that the ability of dispersion hardening to raise the recrystallization temperature, and to improve creep-resistance in the vicinity of the recrystallization temperature, would provide a means of improving the properties of lead-base alloys.

The investigation is reported in three sections, concerned respectively, with: (1) evaluation of the dispersion-hardening characteristics of various elements; (2) studies to determine solid-solution-strengthening elements for use in conjunction with the dispersion-hardening element; (3) phases (1) and (2) having resulted in the selection of nickel and cadmium as the most suitable dispersion-hardening and solid-solution-strengthening elements, respectively, a study was made of the mechanical properties, microstructure, and corrosion-resistance of ternary lead-base alloys containing various additions of nickel and cadmium.

These investigations culminated in the development of a lead-base alloy containing 1·5 per cent. of cadmium and 0·2 per cent. of nickel. The alloy, characterized by good bend fatigue-resistance, good creep resistance over a range of stresses, and high tensile strength, can be cast and fabricated by conventional techniques. It can be satisfactorily extruded or rolled, and can be joined by lead burning.

Preliminary results suggest that, although a slow ingot-solidification rate may adversely affect the alloy's properties, a final annealing treatment will partially overcome these effects.

The resistance of the alloy to sulphuric-acid solutions was found to be comparable to that of copperized lead.

Influence of Pressure on the e.m.f. of Nickel-containing Thermocouple Materials

See abstract on p. 192.

Spectrophotometric Determination of Major Amounts of Cobalt in Nickel Alloys

N. M. SILVERSTONE and B. B. BACH: 'The Spectrophotometric Determination of Major Amounts of Cobalt in Nickel Alloys.'

Metallurgia, 1961, vol. 63, Apr., pp. 205–7.

Major amounts of cobalt in nickel alloys are usually determined either titrimetrically with ferrocyanide, or gravimetrically using 1-nitroso-2-naphthol. In the titrimetric method manganese is known to interfere, and the gravimetric method tends to be unwieldy when dealing with large amounts of cobalt. Nitroso-R-salt is used to form a coloured cobalt complex which may be measured spectrophotometrically, but, owing to the high sensitivity of the reaction, very small sample weights must be used when cobalt is present as a major constituent. The work described was carried out in response to requests for an alternative accurate method for determining large amounts of cobalt in nickel alloys.

The use of E.D.T.A. and hydrogen peroxide for the colorimetric determination of cobalt has been reported: when a faintly acid solution of a cobalt salt is heated to boiling in the presence of E.D.T.A. and hydrogen peroxide, the very stable purple-coloured cobalt III/E.D.T.A. complex is formed. Other metals frequently present in nickel alloys, such as molybdenum, chromium and manganese, were examined under these conditions and it was found that only chromium gave a colour similar to that of cobalt. Attempts fully to suppress the colour due to chromium were not satisfactory and steps were therefore taken to remove this element.

It was found that, in the presence of iron, chromium is precipitated by pyridine as a mixture of ferric and chromic hydroxides and, provided that sufficient iron is present, the precipitate is readily filtered. Titanium, aluminium and zirconium are also precipitated, leaving in solution cobalt, nickel, copper, manganese and molybdenum.

Following the pyridine separation, and treatment with E.D.T.A. and hydrogen peroxide, coloured solutions were obtained with cobalt, nickel and copper. The cobalt is determined spectrophotometrically, and interference due to copper and nickel is eliminated.

The authors give full details of the recommended practice. Data derived from determination of cobalt in nickel-alloy samples ('Nilo K', 'Nimonic 100') show the method to be accurate and to exhibit good reproducibility, with a mean coefficient of variation better than $\pm 0{\cdot}3$ per cent. of content over the range 15–21 per cent. cobalt.

Stress-Corrosion-Cracking Test for Copper-base Alloys

See abstract on p. 199.

NICKEL-IRON ALLOYS

Conference on Magnetism and Magnetic Materials

Papers presented at the *Conference on Magnetism and Magnetic Materials* held in New York in November 1960, under the auspices of the American Institute of Electrical Engineers and the American Institute of Physics, are published in a *Supplement* to the *Journal of Applied Physics*, March 1961. The range of subjects covered is indicated by the section classification employed: ordered spin systems; computer devices; spin configurations; metallic films; nuclear hyperfine fields; ferromagnetic resonance; high coercive materials; exchange interactions and nonlinear microwave processes; magnetization processes and fine particles; anisotropy; domain walls and domain-wall motion; microwave devices; metals and alloys; soft magnetic materials; oxides.

References to papers dealing specifically with nickel-containing materials are listed below.

E. BANKS, N. H. RIEDERMAN, H. W. SCHLEUNING and L. M. SILBER: 'Preparation and Properties of Thin Ferrite Films', pp. 44s–5s.

H. P. LEMAIRE and W. J. CROFT: 'Ferrite Thin Films', pp. 46s–7s.

E. PRINCE: 'Structure of Nickel Chromite', pp. 68s–9s.

D. O. SMITH: 'Anisotropy in Nickel-Iron Films', pp. 70s–80s.

S. CHIKAZUMI: 'Epitaxial Growth and Magnetic Properties of Single-Crystal Films of Iron, Nickel and 'Permalloy' ', pp. 81s–2s.

C. D. GRAHAM and J. M. LOMMEL: 'Magnetic Anisotropies of Nickel Films Evaporated and Measured at 10^{-8} mm. Hg and Below', pp. 83s–4s.

G. P. WEISS and D. O. SMITH: 'Annealing of Oblique-Incidence 'Permalloy' Films', pp. 85s–6s.

M. S. COHEN: 'Anisotropy in 'Permalloy' Films Evaporated at Grazing Incidence', pp. 87s–8s.

A. SEGMULLER: 'Annealing Behaviour and Temperature Dependence of the Magnetic Properties of Thin 'Permalloy' Films', pp. 89s–90s.

R. J. PROSEN, J. O. HOLMEN and B. E. GRAN: 'Rotatable Anisotropy in Thin 'Permalloy' Films', pp. 91s–2s.

T. MATCOVICH, E. KOROSTOFF and A. SCHMECKENBECHER: 'Anisotropy Rotation in Thin 'Permalloy' Films at Room Temperature', pp. 93s–4s.

P. WOLF: 'Free Oscillations of the Magnetization in 'Permalloy' Films', pp. 95s–6s.

M. H. FRANCOMBE and A. J. NOREIKA: 'Some Properties of Uniaxial 'Permalloy' Films Prepared by Cathodic Sputtering', pp. 97s–8s.

E. KAY: 'Magnetic Thin Films Prepared by Sputtering', pp. 99s–101s.

G. K. WERTHEIM: 'Mössbauer Effect: Applications to Magnetism', pp. 110s–17s.

M. WEGER, E. L. HAHN and A. M. PORTIS: 'Transient Excitation of Nuclei in Ferromagnetic Metals', pp. 124s–5s.

R. G. SHULMAN: 'Nuclear Magnetic Resonance in NiF_2 Domain Walls', pp. 126s–8s.

R. F. SOOHOO: 'Excitation and Boundary Effects in Spin-Wave Resonance', pp. 148s–50s.

R. B. CAMPBELL and C. A. JULIEN: 'Structure of 'Alnico V' ', pp. 192s–4s.

H. ZIJLSTRA: 'Magnetic Annealing of 'Ticonal G' Magnet Steel', pp. 194s–6s.

K. J. KRONENBERG: 'Crystal-Lattice Investigation of the Permanent-Magnet Precipitate in Various 'Alnico' Materials', pp. 196s–8s.

J. S. SALLO and K. H. OLSEN: 'Studies of a High-Coercivity Electrodeposit Having a Lamellar Structure', pp. 203s–4s.

C. R. ABELEDO and P. W. SELWOOD: 'Temperature Dependence of Spontaneous Magnetization in Superparamagnetic Nickel', pp. 229s–30s.

W. J. SCHUELE and V. D. DEETSCREEK: 'Preparation, Growth and Study of Ultrafine Ferrite Particles', pp. 235s–6s.

W. W. MALINOFSKY and R. W. BABBITT: 'Fine-Grained Ferrites. I. Nickel Ferrite', pp. 237s–8s.

W. H. MEIKLEJOHN: 'Exchange Anisotropy in Stainless Steel', pp. 274s–5s.

H. W. FULLER, H. RUBENSTEIN and D. L. SULLIVAN: 'Spiral Walls in Thin Magnetic Films', pp. 286s–7s.

F. G. WEST: 'Magnetoresistive Measurements on Domain Rotation and Wall Development in Ni-Fe Alloy Films', pp. 290s–2s.

R. L. COREN and H. J. JURETSCHKE: 'Magnetoresistance and Magnetic Switching in 'Permalloy' Films', pp. 292s–3s.

S. METHFESSEL, S. MIDDELHOEK and H. THOMAS: 'Nucleation Processes in Thin 'Permalloy' Films', pp. 294s–5s.

A. J. P. MEYER and G. ASCH: 'Experimental g' and g Values of Fe, Co, Ni and Their Alloys', pp. 330s–3s.

E. W. PUGH and B. E. ARGYLE: 'Low-Temperature Pyromagnetic Measurements on Nickel', pp. 334s–5s.

P. J. FLANDERS and F. R. L. SCHOENING: 'Precipitation in Gold-Nickel Single Crystals', pp. 344s–5s.

P. K. KOH: 'Cube Texture in Ultra-thin Molybdenum 'Permalloy' Tape', pp. 358s–9s.

E. M. TOLMAN: 'Diffusion Reactions in the Ageing of '4-79 Molybdenum Permalloy' ', pp. 360s–1s.

D. GERSTNER and E. KNELLER: 'High-Temperature Magnetic Lag in Iron-Nickel Alloys', pp. 364s–5s.

J. M. BLANK: 'Equilibrium Atmosphere Schedules for the Cooling of Ferrites', pp. 378s–9s.

R. C. TURNBULL: 'Reinterpretation of the Reaction Kinetics of Nickel Ferrite', pp. 380s–1s.

A. P. GREIFER, Y. NAKADA and H. LESSOFF: 'Preparation and Properties of Low-Loss Ferrites', pp. 382s–3s.

H. F. REMDE: 'Effect of Cobalt Oxide in Porous Nickel Ferrites at Very High Frequency', pp. 384s–5s.

C. J. KRIESSMAN and S. E. HARRISON: 'Cation Distribution in Magnesium-Nickel Ferrites', pp. 392s–3s.

Magnetization of Iron-Nickel Alloys under Hydrostatic Pressure

J. S. KOUVEL and R. H. WILSON: 'Magnetization of Iron-Nickel Alloys under Hydrostatic Pressure.' *Jnl. Applied Physics*, 1961, vol. 32, Mar., pp. 435–41.

Subsequent to their development of an apparatus highly sensitive to magnetization changes produced by pressure, the authors studied the magnetization of iron, nickel and various iron-nickel alloys as a function of magnetic fields of up to 12000 Oe. and hydrostatic pressures of up to 3000 atm. The measurements, which were carried out at room temperature, were intended, in particular, to throw light on the large variations in the saturation magnetization and Curie temperature reported to occur with pressure in iron-nickel alloys containing about 30 at. per cent. nickel.

Assessed in conjunction with the results reported by PATRICK on the pressure dependence of Curie temperatures (see *Physical Review*, 1954, vol. 93, Feb. 1, pp. 384–92), the data are regarded as indicating that σ_o (the saturation moment at 0°K), as well as θ (the Curie temperature), decreases rapidly as a result of compression in f.c.c. alloys containing about 30 per cent. nickel [an effect which is in agreement with the recent low-temperature pressure measurements of KONDORSKY and SEDOV (*ibid.*, 1960, vol. 31S, May, pp. 331s–5s)]. Both σ_o and θ decreased with increase in iron content. When the 30 per cent. nickel alloy was partially transformed to body-centred-cubic, its σ_o and θ rose to much higher values, but its magnetization was much less pressure-dependent. It is concluded that, unlike the more simple ferromagnetic body-centred-cubic alloys, the face-centred-cubic iron-nickel alloys are characterized by a coexistence of ferromagnetic and antiferromagnetic order, similar to that recently proposed for the nickel-manganese alloys.

Anodic-Polarization Behaviour of Iron-Nickel Alloys in Sulphuric-Acid Solutions

See abstract on p. 194.

Spectrophotometric Determination of Major Amounts of Cobalt in Nickel Alloys

See abstract on p. 184.

CAST IRON

Kinetics of Thermally-Activated Transformations in S.G. Iron

J. H. GITTUS: 'Kinetics of Thermally-Activated Transformations in S.G. Iron.'

Iron and Steel, 1961, vol. 34, Apr., pp. 124–30.

About 80 per cent. of all S.G. iron castings are heat-treated before use, mainly as a means of improving ductility and reducing hardness by eliminating eutectic and pearlitic carbides. Where high strengths are required, fine dispersions, designed to inhibit dislocation movement, are produced by tempering martensite and bainite. High-temperature tempering graphitizes these constituents, and tempering at lower temperatures can produce embrittlement (temper-embrittlement). In the present article details are given of the results obtained during a comprehensive investigation of the effects of heat-treatment and composition upon the characteristics of the following reactions: (1) γ+cementite→γ+graphite; (2) γ→martensite, bainite, pearlite; (3) pearlite, bainite, martensite→α+graphite; (4) temper-embrittlement and temper-toughening; (5) precipitation hardening.

Reaction (1) was found to be accelerated by magnesium and retarded by sulphur. Reaction (2) was controlled by major alloying elements such as nickel, manganese, chromium and molybdenum. Reaction (3) was retarded by traces of tin and copper, and accelerated by grain-refinement. Temper-embrittlement (4) occurred at a higher temperature than temper-toughening and both were time-dependent. Precipitation hardening (5) was induced by addition of nickel and aluminium and holding at 400°–500°C.

The practical significance of the results are discussed. The author concludes that 'embrittling eutectic-carbides are easily removed from magnesium-containing iron by γ-range graphitization, a process which occurs much more rapidly than the equivalent reaction in high-sulphur malleable cast irons. However, the presence of chromium and molybdenum drastically delays this reaction, so that even small contents (adventitious or intentional) of these elements should be eschewed. Fortunately, as the work shows, increased hardenability may be obtained (when required) by the simple expedient of raising the nickel and manganese contents, and, providing a suitable relationship between their contents is preserved, eutectic-carbides are not likely to be encountered. Use of these elements minimizes the need for scrap segregation in foundries wishing to produce both ferrite and tempered-martensite structures in different types of casting. Production of the former structure occurs most rapidly when the prior constituent is γ, particularly if trace elements such as tin are likely to be present, but by ferritizing bainite, fine-grained ferrite may be obtained, with consequent reduction in impact-transition temperature. Improvements in impact properties (albeit of smaller magnitude) may also be derived by use of the sub-critical temper-toughening reaction disclosed in the work and earlier studies.

'The final section, detailing the development of precipitation-hardening S.G. irons, is of smaller immediate practical merit, because of the poor castability of the irons so evolved. Nevertheless, from a theoretical standpoint, it is of interest, since such a means of securing strength has previously been neglected (or accorded little interest) in cast irons. The hardening phase appears to comprise a very fine dispersion of Ni-Al.'

Fluidity and Casting Quality of Stainless Steels and 'Ni-Resist' Cast Irons

See abstract on p. 193.

Repair Welding of Cast Iron

G. M. BLANC, E. HINDSON and H. E. Z. GORDON: 'Repair Welding of Cast Iron.'

Welding and Metal Fabrication, 1961, vol. 29, Mar., pp. 104–10.

This review of the factors involved in repair welding grey cast irons may be divided into three sections. In the first, the authors discuss the general metallurgical characteristics of cast irons, and draw attention to the effects of alloying additions. The second is given up to consideration of the welding characteristics of cast irons, and, in particular, to the problems associated with their low ductility and the risk of structural changes during welding. In the last section, the authors refer to the precautions which must be taken in welding cast irons, and comment on the procedures used in, and the advantages and limitations of, the following welding and brazing techniques: oxy-acetylene welding, braze welding, silver brazing, soft soldering, and arc welding. The electrodes suitable for use in arc welding are discussed, and particular attention is paid to the characteristics of nickel and nickel-alloy electrodes (the types most widely used for welding cast iron).

CONSTRUCTIONAL STEELS

Formulae for Calculation of Upper and Lower Critical Temperatures for Low-Alloy Steels

R. A. GRANGE: 'Estimating Critical Ranges in Heat-Treatment of Steels.'

Metal Progress, 1961, vol. 79, Apr., pp. 73–5.

In heat-treating carbon and low-alloy steels, the metallurgist requiring information on Ac_1 and Ac_3

temperatures usually refers to data tabulated in handbooks. Such tables include values representative of each standard type of steel, but do not reveal the variations to be expected from differences in composition among different heats of each grade. Over the years the authors have determined the upper and lower limits of austenite formation for a great number of heats of steel, representing over 50 different compositions. The data obtained have been statistically analyzed to establish the quantitative effect of each of the principal alloying elements, and, as a result, the individual effects have now been combined in the form of empirical formulae which enable the upper and lower critical temperatures of any heat of medium-carbon and low-alloy steel to be calculated from its chemical composition. In this article the authors present and discuss the evolution and limitations of the two formulæ* (see below).

$$A_s(°F.) = 1333 - (25 \times \%Mn) + (40 \times \%Si) - (26 \times \%Ni) + (42 \times \%Cr)$$

$$A_f(°F.) = 1570 - (323 \times \%C) - (25 \times \%Mn) + (80 \times \%Si) - (32 \times \%Ni) - (3 \times \%Cr)$$

In discussing the limitations of the formulae, the authors restrict their application to steel falling within the following compositional limits: carbon 0·3–0·6, manganese 0–2, silicon 0–1, nickel 0–3·5, chromium 0–1·5, molybdenum 0–0·5, per cent. The mean deviation of measured values from calculated values is stated to be 5–6F.°.

Influence of Silicon and Manganese Contents on the Macrostructure of Low-Alloy-Steel Ingots

T. B. SMITH, J. S. THOMAS, R. GOODALL and R. T. ALLSOP: 'An Effect of Composition on the Macrostructure of Ingots of a Low-Alloy Steel.'

Jnl. Iron and Steel Inst., 1961, vol. 198, June, pp. 161–3.

During a series of experiments, in which a study was being made of the effect of casting temperature on the macrostructure of 50-kg. ingots of a 1·5%Ni–1%Cr–0·3%Mo steel, it was observed that the silicon and manganese content of the steel exerted an influence on the ingot structure which could outweigh that of casting temperature.

In the early stages of the work, while the casting technique was still being developed, several ingots were produced which, though otherwise satisfactory, had a silicon content much lower, and a manganese content slightly lower, than intended (i.e., 0·05–0·07 per cent. silicon, compared with an intended 0·2–0·3 per cent.; and 0·25–0·35 per cent. manganese, compared with 0·45–0·65 per cent.). It was noted that the length of the columnar crystals in these ingots was much greater than that in ingots cast at the same temperature but having higher silicon and manganese contents. Photomacrographs included in the paper demonstrate that, whereas the columnar crystals in a normal ingot are about 1·1 in. (2·75 cm.) long and occupy about 80 per cent. of the ingot volume, in a low-silicon low-manganese ingot they are about 2 in. (5 cm.) long and occupy about 99 per cent. of the ingot volume. Subsequent studies confirmed this effect, which, in the present paper, is explained qualitatively in terms of current theories of ingot structure.

Influence of Pre-Straining and Re-Tempering on Quenched-and-Tempered Steel

E. T. STEPHENSON and M. COHEN: 'The Effect of Pre-straining and Retempering on A.I.S.I. Type 4340.'

Trans. Amer. Soc. Metals, 1961, vol. 54, Mar., pp. 72–83.

Several investigators have reported that the tensile properties of as-quenched or tempered steels are greatly influenced by pre-straining and re-tempering: in general, it has been observed that the tensile strength is increased, that the yield/tensile-strength ratio is increased (often to unity), and that the total elongation and reduction-in-area are decreased. The fact that previous studies had included only a limited number of pre-strain/tempering variations suggested the need for a more comprehensive investigation to determine whether an optimum pre-strain/tempering combination could be achieved. Accordingly, in the work now described, a wide range of pre-strain and tempering treatments were evaluated in relation to their effects on the strength, ductility, hardness and microstructure of an A.I.S.I. 4340 steel of the following composition: carbon 0·4, manganese 0·76, silicon 0·31, phosphorus 0·007, sulphur 0·011, nickel 1·74, chromium 0·82, molybdenum 0·25, per cent.

The steel, received as ½-in.-diameter (1·25-cm.-diameter) bar stock, was cut to length, normalized at 1600°F. (870°C.) for 2 hours, stress-relieved at 1200°F. (650°C.) for 2 hours, and air-cooled prior to machining into tensile-test specimens. The specimens were austenitized at 1525°F. (830°C.) for 40 minutes and quenched in oil. The hardened test bars were tempered (pre-tempered) for 1 hour, pre-strained in tension, and tempered again (re-tempered) for 1 hour (they were water-quenched after each tempering operation). Pre-tempering temperatures varied from 400° to 1200°F. (205° to 650°C.), pre-strains ranged from 0 to 3 per cent. (and up to 7 per cent. where ductility permitted), and re-tempering temperatures ranged from room temperature to 1200°F. (650°C.). After treatment, specimens were subjected to tensile tests, hardness tests, metallographic examination, and, in some cases, notched-bar impact tests.

Straining caused an increase in the strength, and a decrease in the ductility, of A.I.S.I. 4340 steel, together with the entrée of a yield point, effects which were enhanced, as the re-tempering temperature was raised, until a maximum was reached in strength and

* To avoid ambiguity the symbols 'As' and 'Af' have been adopted for use in indicating the two temperatures. 'A_s' is defined as the temperature at which a barely detectable amount of austenite forms during prolonged holding at constant temperature, and 'A_f' is the temperature at which the last trace of ferrite transforms to austenite at prolonged isothermal holding. 'A_s' therefore constitutes a 'ceiling' for tempering, and 'A_f' a 'floor' for austenitizing hypoeutectoid steel.

a minimum in ductility. At this stage, the yield/tensile-strength ratio became unity, and the stable elongation (plastic elongation to the maximum load) fell to zero, even though the total elongation was still appreciable. The practical implications of these findings are discussed.

Electron micrographs included in the paper indicate that the carbides in the tempered martensite are dissolved and reprecipitated by the straining and re-tempering, accounting for part of the observed changes in the mechanical behaviour of the steel. The accompanying yield-point phenomena are attributed to the effective locking of dislocations by a redistribution of the carbon and/or carbides.

Corrosion of Low-Alloy and Stainless Steels in Boiling-Water Reactor Systems

D. C. VREELAND, G. G. GAUL and W. L. PEARL: 'Corrosion of Carbon and Low-Alloy Steels in Out-of-Pile Boiling-Water-Reactor Environment.'

Corrosion, 1961, vol. 17, June, pp. 269t-76t.

Stainless steel has been used as the basic material of construction in most high-temperature water-cooled nuclear-power reactors, primarily to minimize corrosion problems and the difficulties associated with the passage of corrosion products into the recirculating water system. Substitution of carbon steel or low-alloy steel for stainless steel in high-temperature primary-coolant-system applications would, if feasible, offer economic and engineering advantages. Corrosion-resistance, a prerequisite for such applications, is, however, a prime difficulty in this connexion. The extensive tests described by the authors were conducted to study the corrosion behaviour of carbon steels, low-alloy steels, and (for the purpose of comparison) stainless steels in a dynamic test loop simulating the various environments found in a boiling-water-reactor system.

The following types of steel were selected for study: carbon steels; high-strength low-alloy steels ('Triten' and 'Corten'); alloy steels (A.S.T.M. A-302 Grade B, A.I.S.I. 4130, A.I.S.I. 4340, 'T-1'); various grades of chromium, chromium-manganese-nitrogen, chromium-manganese-nickel-nitrogen and chromium-nickel stainless steels (including the precipitation-hardenable steel 'Stainless W'). Dual specimens (comprising carbon steel or low-alloy steel welded to Type 304L 18-8 stainless steel) were also evaluated. The steels were exposed, at 545°F. (285°C.) and 1000 p.s.i., to saturated steam, saturated water and a steam/water mixture. Water and steam conditions were based on an oxygen concentration of 10-20 p.p.m. in the steam with a 1/8 stoichiometric ratio of hydrogen to oxygen (i.e., conditions representative of the hydrogen and oxygen formed in a boiling-water reactor from radiolytic water decomposition). The pH of the water (which was of high purity) was maintained at 7. Details are given of the test facilities and operating conditions, and the results of the corrosion tests (including data on corrosion products released into the system) and of metallographic examinations of the specimens are fully presented.

No significant difference was noted between the corrosion rates of the carbon steels, high-strength low-alloy steels and alloy steels, although all showed higher rates than the A.I.S.I. Type 300 series of stainless steels studied. No selective attack was noted on welded specimens.

The corrosion rates for carbon and low-alloy steels were lower than those obtained by other investigators on similar materials in test loops simulating pressurized-water-reactor systems operated at high pH (>10) with only hydrogen gas in the water. The iron-to-system rates in the present tests were considerably less than those established in similar tests in high-pH and neutral-pH systems with hydrogen but no oxygen in the water.

HEAT- AND CORROSION-RESISTING MATERIALS

Nickel-containing Materials for Use under Severe Service Conditions

E. G. HOLMBERG: 'New Alloys for Extreme Conditions.'

Chemical Engineering Progress, 1961, vol. 57, Apr., pp. 74-8.

In this paper the author comments on the properties and advantages offered by materials which have been developed for use under service conditions which have proved 'extreme' for those previously available. The classes and specific alloys whose performance is discussed, and for which data are presented, are noted below.

(1) *Nickel-containing Steels for Low-Temperature Service.*

The suitability of 2¼, 3½ and 9 per cent. nickel steels and 18-8 type stainless steels is briefly considered.

(2) *Nickel-Chromium-Iron Alloys*

Discussed are:

A 42Ni-30Cr-26Fe alloy ('Incoloy 804'), which exhibits exceptionally good resistance to carburization and oxidation, and offers considerably better resistance to sulphidation than other alloys of about the same nickel content.

A 60Cr-40Ni cast alloy exhibiting improved resistance to fuel-oil-ash corrosion and currently in use as a material for non-cooled superheater supports in U.S. Navy ships burning fuel oil.

A 65Ni-35Cr wrought alloy displaying a resistance to nitric acid considerably higher than that of the austenitic stainless steels.

A 42Ni-21Cr-3Mo alloy (nominal composition: nickel 42, chromium 21, molybdenum 3, manganese 1 max., copper 1·75, carbon 0·05 max., titanium 1, silicon 0·5 max., per cent., remainder iron) having a satisfactory resistance to a variety of corrosive solutions too severe for the A.I.S.I. 300 series of austenitic stainless steels. (The alloy is stated to be exceptionally

resistant to the reducing conditions encountered in sulphuric- and phosphoric-acid solutions and to offer a useful resistance to a variety of oxidizing chemicals.)

(3) *Nickel-Chromium-(Cobalt) Alloys*

An age-hardenable vacuum-melted alloy (containing nickel 50, cobalt 11, chromium 19, molybdenum 10, iron 5 max., titanium 3·15, aluminium 1·65, per cent.) is referred to as exhibiting outstanding strength in the range 1200°–1800°F. (650°–980°C.), and a 78Ni–16Cr–3Al–0·02C alloy exhibiting exceptional oxidation-resistance at temperatures up to 2400°F. (1315°C.) is deemed of particular interest as a cladding material for molybdenum-base alloys subject to catastrophic oxidation at high temperatures.

(4) *Precipitation-Hardening Chromium-Nickel Steels*

(5) *Titanium*

(6) *Zirconium*

(7) *Molybdenum.*

Influence of Heat-Treatment on the Nature and Morphology of Phases Present in Age-Hardenable Nickel-Chromium-Titanium-Aluminium Alloys

E. A. FELL: 'The Effect of Thermal Treatment on the Constitution of 80-20 Nickel-Chromium Alloys Hardened with Titanium and Aluminium.'

Metallurgia, 1961, vol. 63, Apr., pp. 157–66.

The fundamental hardening mechanism for 80-20 nickel-chromium alloys rendered precipitation-hardenable by additions of titanium and aluminium (i.e., the system on which the 'Nimonic' series of high-temperature alloys is based) arises from precipitation of the γ' phase, but BETTERIDGE and FRANKLIN (see *Jnl. Inst. Metals*, 1956–57, vol. 85, July, pp. 473–9: abstract in *Nickel Bulletin*, 1957, vol. 30, No. 7–8, pp. 129–30) have shown that precipitation of chromium-rich carbides determines the amount of creep extension occurring before fracture, and since a critical balance between creep-resistance and ductility is essential to the success of a high-temperature alloy, precipitation of carbides has a significant bearing on stress-rupture properties. In the investigation now reported, the occurrence and distribution of the precipitated phases present in age-hardenable 80-20 nickel-chromium-base alloys were studied, using X-ray-diffraction and optical- and electron-microscopical techniques, within the scope of a general programme of research on the constitution and morphology of phases present in nickel-base high-temperature alloys.

In the paper, consideration is first given to data obtained in determining the effects of varying the time and temperature of heat-treatment. The modifications in structure resulting from varying the titanium, aluminium and carbon contents of the alloys are discussed, and information derived from examination of quenched and creep-tested specimens is presented in illustration of the effects of strain on the kinetics of precipitation. Finally, the influence of heat-treatment on the creep and stress-rupture properties of 'Nimonic 80', as determined by BETTERIDGE and FRANKLIN (*loc. cit.*), is discussed in relation to the observations made during the structural studies now reported. The authors emphasize that no attempt was made to achieve the degree of homogeneity usual in constitutional studies, since it was intended that the alloys should approximate as closely as possible to those used in commercial applications. The reactions which occurred therefore took place over a wider range of temperature than would have been the case for alloys of higher homogeneity.

Five precipitates were identified: titanium carbonitride (Ti(C,N)), the chromium carbides Cr_7C_3 and $Cr_{23}C_6$, and two Ni_3Al-type phases. The data presented are regarded as suggesting that the useful properties of 'Nimonic 80' and related alloys arise from a strengthening of the matrix by general precipitation of the $Ni_3(Ti,Al)$ phase, combined with the formation, at the grain boundaries, of zones sufficiently weak to permit relaxation of local stresses. These zones are induced in the alloys by initial precipitation of Cr_7C_3 phases at the grain boundaries, and are maintained by the subsequent carbide reaction $Cr_7C_3 \rightarrow Cr_{23}C_6$.

On the basis of the work described the authors draw eight main conclusions:

'(1) Of the five minor phases observed, $Cr_{23}C_6$, $Ni_3(Ti,Al)$, and possibly the second Ni_3Al-type phase, precipitate within the grains in an orientation identical with that of the matrix.

'(2) In an alloy which has undergone a typical solution-treatment, the first chromium carbide to precipitate during subsequent heat-treatment in the range 500°–1150°C. is of the Cr_7C_3 type, and, on further heat-treatment, at temperatures below 1050°C., this phase is progressively replaced by a carbide of the $Cr_{23}C_6$ type.

'(3) The carbide reaction, $Cr_7C_3 \rightarrow Cr_{23}C_6$, is accelerated by internal strain, by low carbon content, or by high total titanium plus aluminium content: it is retarded by pre-existing Cr_7C_3 phase, by high carbon, or by low total titanium plus aluminium content.

'(4) The solution temperature of the $Cr_{23}C_6$ phase rises with increase in total titanium plus aluminium content of the alloy.

'(5) The marked development of $Cr_{23}C_6$ precipitate on dislocation sites near the grain boundaries, or as cellular precipitate at grain boundaries, is associated with the presence of internal stress.

'(6) The lattice parameter and the rate of growth of the $Ni_3(Ti,Al)$ phase increase with the titanium: aluminium ratio of the alloy, and with rise in the temperature of precipitation. It is independent of the total titanium plus aluminium content of the alloy.

'(7) Water-quenching from the solution temperature prevents $Ni_3(Ti,Al)$ precipitation during cooling, and increases the rate of growth during subsequent ageing, but the particle size ultimately attained is similar to that of an air-cooled and aged alloy.

'(8) There is no evidence to suggest that the $Cr_{23}C_6$ phase, in either normal or cellular form, has a deleterious effect on the fracture mechanism of the alloys.'

'DCM' Alloy: Microstructural Study

J. E. WILSON and J. F. RADAVICH: 'Phase Identification in Nickel-Base Alloys.'

Metal Progress, 1961, vol. 79, May, pp. 94–6.

The high-temperature alloy 'DCM'* was developed in 1957 for use in turbine blades operating at temperatures up to 1800°F. (980°C.). In this article the findings are presented of a study initiated to identify the phases present in 'DCM': much of the information given is, however, deemed applicable to 'other cast and wrought nickel-base alloys containing aluminium, titanium, molybdenum, chromium and boron'.

The studies were conducted, using optical and electron microscopy and X-ray diffraction techniques, on specimens of different grain-size taken from four test bars which had been subjected to rupture testing and from three vacuum-cast ingots in the as-cast or heat-treated condition. The authors' comments on the results of these studies are supplemented by photomicrographs exemplifying the structures observed.

The 'DCM' matrix comprises a nickel-base solid solution. The microstructural studies indicate that, besides γ' (Ni_3Al), the complex boride (M_3B_2), and a titanium carbonitride phase (Ti(C,N)) are present in the as-cast alloy. All of the phases are found at the grain boundaries; γ' is, however, most prevalent in the matrix, and the other phases in the boundary regions.

Upon heat-treatment or testing at elevated temperatures, another carbide phase ($M_{23}C_6$) forms at the expense of the carbonitride. That fine-grained specimens, which exhibit a substantially shorter rupture life than coarse-grained material, also contain more $M_{23}C_6$ is deemed of significance: it is suggested that the short rupture life probably results from microcracks which initiate in fine-grained material at regions where $M_{23}C_6$ or agglomerated γ' phase are concentrated in the grain boundaries. Fine-grained specimens were also found to be less resistant to intergranular oxidation.

Studies of cross-sectioned bars, carried out to determine phase distribution near the outer surfaces of specimens which had been heat-treated or rupture-tested, revealed that, irrespective of grain-size, the structure changes, from that of the basis metal, to a depleted zone of Ni_3Al, a layer of alpha Al_2O_3, and, on the surface, scales of $(Cr, Fe)_2O_3$ and spinel oxide. The presence of the alpha Al_2O_3 layer was anticipated in view of the zone of depleted Ni_3Al, but little is known of the effect of the alpha Al_2O_3 on the resistance of this alloy system to high-temperature oxidation.

'IN-100' Nickel-base Casting Alloy for Use at 1900°F.

C. G. BIEBER and T. E. KIHLGREN: 'A New Cast Alloy for Use at 1900°F.'

Metal Progress, 1961, vol. 79, Apr., pp. 97–9.

'IN-100', the properties of which are reviewed in this article, is the latest in a series of cast turbine-blading alloys originating in the introduction of 'Inconel 713C' in 1956. Intended for service at temperatures up to 1900°F. (1035°C.), the alloy is melted and investment-cast *in vacuo* and is at present used in the as-cast condition.

Compositional limits are: cobalt 13–17, chromium 8–11, aluminium 5–6, titanium 4·5–5·5, molybdenum 2–4, vanadium 0·7–1·2, carbon 0·15–0·2, boron 0·008–0·02, zirconium 0·03–0·09, per cent., remainder nickel. Iron is limited to a maximum of 1 per cent., and the combined aluminium and titanium additions should fall within the range 10–11·5 per cent.

Tensile strength (140,000 p.s.i. (62·5 t.s.i.: 98·5 kg./mm.²)), and yield strength (122,000 p.s.i. (54·5 t.s.i.: 86 kg./mm.²)) are stated to remain unchanged up to 1400°F. (760°C.), but then begin to decrease. Elongation values at temperatures up to 2000°F. (1095°C.) vary from 6 to 8 per cent.

Specifications covering experimental production require a minimum rupture life of 23 hours at 1800°F. (980°C.) and a stress of 29,000 p.s.i. (13 t.s.i.: 20·5 kg./mm.²), with 4 per cent. minimum elongation. These requirements are stated to be easily met, and lives of between 29 and 43 hours, with 7–9 per cent. elongation, have been reported for production heats.

Resistance to thermal shock is comparable to that of 'Inconel 713C', and the data available to date indicate that the alloy exhibits adequate oxidation-resistance for service in jet engines operating at 1850°–1900°F. (1010°–1035°C.). For longer periods of operation or higher service temperatures, some type of protection, such as aluminizing or chromizing, may, it is considered, be needed.

Investment castings in a number of commercial configurations have been successfully produced, but where castings are subjected to extreme restraint during solidification, some tendency to hot tearing has been observed: in such cases, it is recommended that boron and zirconium should be kept on the low side of the specification.

Stress-rupture curves for 'IN-100' are included in the article, and other data permit comparison of its rupture strength at 1500°, 1600° and 1800°F. (815°, 870° and 980°C.) with those of various wrought and cast commercial high-temperature alloys.

Properties of 'AM-355' Steel in Forgings and Bar Stock

P. A. BERGMAN: ' 'AM-355' for Gas-Turbine Engines.'

Metal Progress, 1961, vol. 79, May, pp. 79–85.

Small gas-turbine engines developed for missiles, aircraft and helicopters are finding application in the marine and industrial fields, and, as operating conditions become more severe, some of the materials originally used for highly stressed rotating parts in the compressor section are being replaced by those capable of satisfying the demands for higher strength

* Composition: carbon 0·08 max., manganese 0·1 max., silicon 0·15 max., sulphur 0·015 max., chromium 14-16, titanium 3·35-3·65, boron 0·07-0·09, aluminium 4·4-4·8, molybdenum 4·5-6, iron 4-6, copper 0·1 max., per cent., remainder nickel.

and corrosion-resistance. The controlled-transformation stainless steel 'AM-355'* offers promise of meeting these requirements, and is being used for such compressor parts as blades, discs, spool rotors, spacers, seals and shafts (the blades are rolled from bar stock, and the other parts are machined from forgings). In this article the author, who is associated with the Small Aircraft Engine Department, General Electric Company, Mass., discusses the general characteristics and properties of 'AM-355' forgings and bar stock, and the advantages the steel offers in gas-turbine applications.

From the point of view of composition and corrosion-resistance 'AM-355' occupies a position intermediate between the A.I.S.I. 400 series of martensitic stainless steels and the 300 series of austenitic stainless steels. In the annealed condition its structure is austenitic; in the heat-treated condition, martensitic. Its resistance to stress corrosion and its good notch ductility enable heat-treated material to be used at higher hardnesses than, for example, Type 403 12 per cent. chromium steel.

After outlining the heat-treatment cycle developed for the steel and alluding to the mechanical properties obtainable by suitable heat-treatment, the author comments on data illustrating the resistance of the steel to salt spray and stress corrosion. Data exemplifying tensile, stress-rupture, fatigue and impact properties are presented, and reference is made to the results of temperature-cycling tests carried out to determine whether the steel possessed the high degree of alloy stability needed to maintain clearances and rotor balance and to prevent microstructural changes which might result in embrittlement.

In initial applications use of air-melted material gave rise, due to segregation of chromium, nickel and molybdenum at the centre and top of larger ingots, to the problem of retained austenite (with its deleterious effects on creep properties). Homogenizing treatment proved of no avail, but, eventually, introduction of consumable-electrode melting, and adjustment of forging temperature and heat-treatment procedures minimized the amount of austenite retained. A rapid macroetch test facilitated control over the homogeneity of the stock. The specimen is immersed for 20–30 seconds in an aqueous solution of 40 per cent. hydrochloric acid and 20 per cent. nitric acid at 160°F. (70°C.). If any white areas are observed, the material is rejected due to excessive retained austenite.

* Nominal composition: carbon 0·12, chromium 15·5, nickel 4·5, molybdenum 3, nitrogen 0·1, per cent.

Properties of Nickel/Aluminium-Oxide and Nickel-Aluminide/Aluminium-Oxide Sintered Materials

W. JELLINGHAUS: 'Sintered Materials Produced from Nickel or Nickel Aluminide and Aluminium Oxide.' *Archiv f. d. Eisenhüttenwesen*, 1961, vol. 32, Mar., pp. 187–97.

Compositions comprising a high volume percentage of a hard phase of low plasticity and thermal conductivity distributed throughout a soft metal of higher ductility have found wide use as tool materials. The extension of this concept to the high-temperature field has not, however, so far met with success. Nickel aluminide has attracted attention as a material offering promise for high-temperature applications, and the studies outlined in the present paper were carried out to evaluate its properties further, and, in particular, to obtain information on the high-temperature properties of powder-metallurgically-produced materials comprising nickel or nickel aluminide dispersion strengthened with aluminium oxide. Emphasis was placed on the effects of hard-particle contents below 66 vol. per cent.

A fine-grained powder consisting of a mixture of α-Al_2O_3 and metallic nickel was produced by oxidizing finely-ground nickel-aluminium alloys (containing 66–75 at. per cent. aluminium) in water. This powder was employed, with carbonyl-nickel powder, to produce sintered Ni/Al_2O_3 material for testing. The properties of NiAl and $NiAl/Al_2O_3$ specimens produced by sintering compacted powder mixtures were also evaluated. Finally tests were carried out to obtain data on the high-temperature properties of $NiAl/Al_2O_3$ specimens in which the aluminium-oxide dispersion was achieved by heating, and thus oxidizing, porous nickel-aluminide specimens in air (either after sintering, or after compacting and before sintering).

Full details are given of the powder-metallurgical techniques and the test conditions employed in the study.

The test data presented show that Ni/Al_2O_3 sintered materials can be rolled at aluminium-oxide contents up to 8 wt. per cent: a sintered specimen of this type containing somewhat more than 50 vol. per cent. Al_2O_3 exhibited a bend creep strength of 18 kg./mm.^{2} at 900°C. in 100-hour tests.

The flow rate of sintered nickel-aluminide specimens at temperatures in the region of 800°C. was found to be considerably decreased by incorporation of fine-grained aluminium oxide, and, at 40 vol. per cent. of aluminium oxide, a 100-hour bend strength of 28 kg./mm.^{2} was achieved at 800°C.

Oxidation of porous sintered nickel-aluminide specimens at temperatures between 900°C. and 1200°C. resulted in the formation of aluminium oxide and an increase in the nickel content of the metal phase. At an increase in weight of 11–15 per cent., the distribution of the oxide and metal phase was associated with bend strengths at 800°C. of up to 28 kg./mm.^{2} after more than 100 hours. The structure was finer, and creep strength somewhat better, when the specimens were oxidized at 550° and 650°C. to achieve a 7 per cent. increase in weight after compacting and before sintering.

The modulus of elasticity of nickel aluminide (containing 50 at. per cent. nickel) was increased, by incorporation of 40 vol. per cent. of Al_2O_3, from 8000 kg./mm.^{2} to 14,200 kg./mm.^{2}.

Oxidation Behaviour of Nickel-Aluminium Alloys

K. FUEKI and H. ISHIBASHI: 'Oxidation Studies on Ni-Al Alloys.'

Jnl. Electrochemical Soc., 1961, vol. 108, Apr., pp. 306-11.

The investigation reported formed part of a series undertaken to throw light on the oxidation characteristics of binary alloys at high temperatures (a paper reporting previous work on nickel-cobalt alloys was abstracted in *Nickel Bulletin*, 1961, vol. 34, No. 2, p. 41).

Specimens of pure nickel and four nickel-aluminium alloys (containing, respectively, 1·02, 2·95, 7·07 and 12 mole per cent. of aluminium) were selected for study. Using a vacuum microbalance, oxidation rates were determined at temperatures in the range 700°-900°C. The oxide films formed were examined by metallographic, X-ray-diffraction and electron-diffraction techniques, and films stripped from the basis alloys were subjected to quantitative chemical analysis to discover the amounts of nickel and aluminium oxidized. The change in the composition of the alloy surface was investigated by measuring the lattice parameters of the surface before and after oxidation.

The data derived from each phase of the investigation are fully presented. The weight gain of all the specimens was found to follow the parabolic rate law. The outer layer of the oxide film consisted of only NiO; the aluminium compounds present in the inner layer comprised γ-Al_2O_3 and/or $NiO{\cdot}Al_2O_3$.

The data show that addition of small amounts of aluminium improves the oxidation-resistance of nickel at 700°C., an effect which is considered to be due to the formation of an adherent and protective γ-Al_2O_3 film on the alloy. At 900°C., however, the oxidation rate of nickel was reduced to a lesser degree by the addition of aluminium, and this result is attributed to the formation of the porous $NiO{\cdot}Al_2O_3$ film, which cannot effectively suppress the flow of nickel ions. A close relationship appeared to exist between the formation of the porous $NiO{\cdot}Al_2O_3$ film and subscale formation, and, accordingly, a close relationship is deemed to exist between subscale formation and the effects of aluminium additions on the oxidation of nickel.

Influence of Pressure on the e.m.f. of Nickel-containing Thermocouple Materials

F. P. BUNDY: 'Effect of Pressure on e.m.f. of Thermocouples.'

Jnl. Applied Physics, 1961, vol. 32, Mar., pp. 483-8.

Although thermocouples have proven to be the most satisfactory indicators of temperature in internally heated super-pressure cells, the absolute accuracy of the readings has been in question because of the lack of information on the effect of pressure on the thermal e.m.f. of thermocouples at the pressures now being attained. In the experiments described, the pressure e.m.f.'s of the following common thermocouple materials were measured for a ΔT of about 100°C. at pressures in the range 0 to 72 kbar: 'Constantan', 'Alumel', 'Chromel', platinum, nickel, copper, platinum-10 per cent. rhodium, nickel -18 per cent. molybdenum. The experiments were designed around the 'belt' super-pressure apparatus, development of which offered a means of attaining pressures of up to 100 kbar and internal temperatures greater than 2000°C.

Pressure thermal e.m.f.'s are plotted. 'Constantan' showed the strongest effect and platinum the next strongest. Nickel, 'Alumel' and the platinum-10 per cent. rhodium alloy all gave about the same effect. Copper gave a weak positive effect, and 'Chromel' and the nickel-18 per cent. molybdenum alloy were both weakly negative.

Corrections for pressure are deduced for common thermocouples made up of pairs of the materials studied. A number of thermocouple pairs were compared at temperatures up to 1200°C. and pressures up to 58 kbar. Below 200°-300°C. the deviations between them agreed quite well with the absolute data on single metals taken at ΔT of 100°C. At higher temperatures the deviations diminished and generally reversed. The results are considered to suggest that the deviation δT of the readings of two thermocouples at a given pressure follows roughly the relationship

$$\delta T = A(P)\Delta T + B(P)\Delta T^2,$$

where ΔT is the temperature interval in the pressurized zone, $A(P)$ and $B(P)$ are functions of pressure (roughly linear), and $B(P)$ is generally opposite in sign to $A(P)$ and is large enough to dominate the $A(P)$ term at higher ΔT's.

Determination of Cobalt in High-Temperature Alloys

H. KIRTCHIK and F. H. SWEARINGEN: 'The Determination of Cobalt in Heat-Resistant Alloys.'

Analyst, 1961, vol. 86, Mar., pp. 188-95.

The analytical technique described is stated to be applicable to the determination of cobalt in a wide range of high-temperature alloys, and to be suitable for umpire purposes.

The specific precipitation of cobalt by a combination of phenylthiohydantoic and thioglycollic acids is used to overcome the difficulties associated (due to interference from many elements) with procedures previously available. The precipitated cobalt is accompanied only by trace amounts of iron and nickel, which offer no hindrance to the subsequent direct potentiometric titration with potassium-ferricyanide solution in the presence of ethylenediamine.

The element has been successfully determined in iron-, cobalt-, and nickel-base alloys containing various combinations of aluminium, chromium, copper, iron, manganese, molybdenum, nickel, niobium, silicon, tantalum, titanium, tungsten, vanadium and zirconium, and, for alloys containing more than 3 per cent. cobalt, consistent results (with errors of less than 2-4 per cent.) can, it is claimed, be

expected. Use may be made of a standard potentiometric-titration apparatus or a pH meter with platinum and calomel electrodes.

Spectrophotometric Determination of Major Amounts of Cobalt in Nickel Alloys

See abstract on p. 184.

Spectrophotometric Determination of Boron in Steel and High-Temperature Alloys

W. L. KARPEN: 'Spectrophotometric Determination of Boron in Steel and High-Temperature Alloys.'
Analytical Chemistry, 1961, vol. 33, May, pp. 738–40.

The author gives details of a spectrophotometric procedure which, based on the rose-coloured boron tetrabromochrysazin complex, is applicable to the determination of boron in steels and high-temperature alloys in amounts ranging from 0·0002 to 0·025 per cent. The specimen is dissolved in a mixture of perchloric acid and phosphoric acid, and the boron is distilled as the trimethyl boron ester. (In the presence of phosphoric acid, the perchloric acid does not esterify the methanol.) The boron and reagent readily combine in 96 per cent. sulphuric acid in a 1 to 1 mole ratio. The coloured complex reaches maximum intensity in one hour, and the absorbance is measured at 540 mμ.

Data derived from analysis of the nickel-chromium-cobalt alloy 'Waspaloy' are included in the paper in illustration of the results obtained by the technique.

Copper-Nickel-Iron Alloy for Feedwater-Heater Tubing

'Cheaper Tube for Feedwater Heaters.'
Chemical Engineering, 1961, vol. 68, May 1, pp. 114, 116.

This note draws attention to the availability and properties of a copper-nickel-iron alloy (nominal composition: copper 64·15, nickel 30, iron 5·25, manganese 0·6, per cent.) stated to retain its strength at high temperatures, and to exhibit excellent resistance to stress-corrosion cracking, and high resistance to corrosion by sea water. The physical properties of the alloy are listed below.

Density	0·320 lb./cu.in.
Modulus of elasticity	22,000,000 p.s.i.
Thermal conductivity (at 68°F.)	14 Btu./(hr.) (sq.ft.) (°F./ft.)
Coefficient of expansion, linear (77°-572°F. av.)	9×10^{-6} (ft./ft.)/°F.

Heat-exchanger tubing in the alloy is produced by Anaconda American Brass Company in the annealed state (which facilitates its fabrication into U bends). The tubing can be welded into steel tube sheets using procedures similar to those for other copper-nickel alloys, but, for optimum strength, use of 'Monel' filler metal is recommended. The following tensile strengths are stated to be typical for tubing with 0·049 in. (1·24 mm.) wall: $\frac{5}{8}$-in. (1·5-cm.) O.D. tubing: 84,700 p.s.i. (37·8 t.s.i.: 59·5 kg./mm.²); $\frac{3}{4}$-in. (1·8-cm.) tubing: 88,000 p.s.i. (39·3 t.s.i.: 62 kg./mm.²). The maximum permissible working stress in tension at 600°F. (315°C.) is 15,200 p.s.i. (7 t.s.i.: 10·5 kg./mm.²).

Stress-Rupture Properties of Type 304 18-8 Stainless Steel

W. D. JENKINS, W. A. WILLARD and W. J. YOUDEN: 'Stress-Rupture Tests at 1350°F. on Type 304 Stainless Steel.'
Materials Research and Standards, 1961, vol. 1, Feb., pp. 104–8.

The tests described in the paper were carried out, at the request of the Test Methods Panel of the A.S.T.M.-A.S.M.E. Joint Committee on the Effect of Temperature on the Properties of Metals, to determine the suitability of a heat of Type 304 stainless steel as a source for calibration specimens for stress-rupture testing.

In a test programme statistically designed to obtain a maximum amount of data from a limited number of tests, specimens selected to evaluate the uniformity of the bars processed from the heat were subjected, at 1350°F. (730°C.), to a stress (13,500 p.s.i. [6 t.s.i.: 9·5 kg./mm.²]) giving an average life of 100 hours. The test specimens (obtained from bar processed from four ingots) were identified in terms of ingot, and, in some cases, position in the ingot.

Detailed statistical analysis of the test data revealed a standard deviation of 20 hours in stress-rupture life.

Fluidity and Casting Quality of Stainless Steels and 'Ni-Resist' Cast Irons

A. M. PATTON: 'The Fluidities of Some Cast Stainless Steels and 'Ni-Resist' Cast Irons.'
Brit. Foundryman, 1961, vol. 54, May, pp. 208–11.

Although substantial tonnages of stainless-steel castings are produced annually, the literature contains few data on fluidity and general casting properties. In the investigation described, these properties were studied in relation to various commercial grades of cast stainless steels and two experimental stainless steels of 18–10 type modified so as to improve fluidity and casting qualities. In certain applications 'Ni-Resist' cast irons can be considered as alternative materials of construction to stainless steels, and two common grades of the iron were therefore included within the scope of the investigation.

Fluidity and casting quality were assessed using a spiral test. A quick-reading immersion thermocouple was employed to measure the metal temperature immediately before the casting of each spiral, and the performance of each material was evaluated on the

Compositions of Materials Subjected to Fluidity Tests
(*See abstract on p.* 193)

Alloy	C %	Cr %	Si %	Mn %	P %	S %	Ni %	Nb %	Fe %	Other Elements
'Ni-Resist I'	2·91	2·2	2·15	1·20	0·024	0·011	15·8	—	bal.	Cu 7·12
'Ni-Resist II'	2·63	2·2	2·00	1·25	0·024	0·010	20·2	—	bal.	—
13% chromium stainless steel	0·255	13·8	0·7	0·4	0·025	0·013	0·4	—	bal.	—
18–10 stainless steel	0·049	18·0	0·4	0·3	0·021	0·011	9·95	0·59	bal.	—
18–10 stainless steel	0·101	18·1	0·4	0·4	0·017	0·012	9·82	1·05	bal.	—
18–8 stainless steel	0·102	18·1	0·7	0·4	0·017	0·012	7·95	1·10	bal.	—
25–15 stainless steel	0·118	15·6	0·73	0·5	0·012	0·005	24·95	1·18	bal.	—
3% silicon stainless steel	0·138	18·5	3·07	0·6	0·012	0·006	10·02	1·18	bal.	—
3% aluminium stainless steel	0·120	17·8	1·07	0·7	0·015	0·005	9·98	1·10	bal.	Al 2·86

basis of two criteria: fluidity was measured in terms of the length of the spiral run, and casting quality in terms of that percentage of the spiral casting over which the molten metal had completely filled the top corners of the mould. The compositions of the materials studied are given in the table above.

The data presented are regarded as establishing, on a quantitative basis, the superiority of the fluidity and casting quality (irrespective of whether these properties were assessed in terms of pouring temperature or superheat) of the 'Ni-Resist' cast irons to those of the cast stainless steels. Of the conventional corrosion-resisting steels, the 18–10 grade with the lower carbon level (0·05 per cent.) appeared to be the best casting material. Its casting quality was the most satisfactory of the steels tested, and any advantage which the higher-carbon (0·01 per cent.) grade might appear to offer, due to its better fluidity, was more than outweighed by the higher-impact resistance of the former steel. The fluidity and casting quality of the stainless steels were improved by increase in nickel content, and, in the case of the 18–10 steel, addition of 3 per cent. silicon or 3 per cent. aluminium resulted in a marked improvement in casting quality and, in the case of the silicon addition, also in fluidity.

Critical pH and Critical Current Density for Passivity in Metals

H. H. UHLIG: 'Critical pH and Critical Current Density for Passivity in Metals.'

Jnl. Electrochemical Soc., 1961, vol. 108, Apr., pp. 327–30.

In the introductory section of this paper the author refers, as a characteristic of passive metals as at present considered, to the fact that their corrosion potentials in the passive state are more noble than their activation or Flade potentials, the latter marking the potential which divides the active from the passive region. In general, hydrogen-ion activity is one of the variables in a given aqueous environment determining passivity, and a critical pH exists which marks the boundary between stable and unstable passivity. The present paper is concerned with the relationship between critical pH and Flade potential, and the evaluation of critical pH on the basis of knowledge of polarization and potential behaviour.

The following equation (where K and λ are constants) is derived for the relation of pH to the critical current density necessary to passivate metals: i (crit.) = $K(H^+)^{\lambda}$. Accordingly, a linear relation between log i (crit.) and pH is observed for chromium, nickel and 14 per cent. chromium–iron alloys. When passivity occurs by reason of dissolved oxygen, it is shown that i (crit.) usually equals the limiting diffusion current density for O_2. A critical pH exists above which passivity is stable, but not below. This critical pH decreases linearly with the logarithm of dissolved O_2 concentration. Calculated and observed values for the critical pH of 14 per cent. chromium–iron alloys are in reasonable agreement.

In the absence of O_2, passivity is achieved when the rate of reduction of H^+ exceeds i (crit.). Metals in this category must have Flade potentials more active than the hydrogen electrode in the same solution. More active Flade potentials accompany greater thermodynamic stability of the passive film, accounting in part for greater resistance to breakdown of passivity by specific anions. Among the metals in this category achieving high corrosion-resistance in deaerated acids are titanium, tantalum, zirconium and molybdenum.

Anodic-Polarization Behaviour of Iron-Nickel Alloys in Sulphuric-Acid Solutions

G. ECONOMY, R. SPEISER, F. H. BECK and M. G. FONTANA: 'Anodic-Polarization Behaviour of Iron-Nickel Alloys in Sulfuric-Acid Solutions.'

Jnl. Electrochemical Soc., 1961, vol. 108, Apr., pp. 337–43.

In this paper the authors discuss the data obtained in studies (based on galvanostatic and potentiostatic

techniques) of the anodic behaviour of nickel, iron and ten iron-nickel alloys (with iron contents ranging from 10 to 95 at. per cent.) in hydrogen-saturated sulphuric-acid solutions at 25°C.

Anodic behaviour is correlated with metallurgical structure and electrochemical environment, and particular emphasis is placed on the interpretation of corrosion potential, passivation current, passive current, and the passive/active transition. The data demonstrate that all the alloys become passive at noble potentials, and that, in general, the behaviour of the alloys was intermediate between that of iron and nickel.

Diffusion of Niobium with Nickel and Stainless Steel

L. S. BIRKS and R. E. SEEBOLD: 'Diffusion of Nb with Cr, Fe, Ni, Mo, and Stainless Steel.'

Jnl. Nuclear Materials, 1961, vol. 3, Mar./Apr., pp. 249–59.

Because of its low nuclear cross section and relatively small mass-transfer, niobium is of current interest as a container for liquid-metal heat exchangers. Its poor oxidation-resistance at elevated temperatures, however, necessitates protective cladding, usually with stainless steel. Since intermetallic diffusion often results in cracking between the niobium and the cladding material, it was thought that a study of the binary diffusion of niobium with the main constituents of stainless steel (chromium, iron, nickel, molybdenum) might yield data of value in predicting alloy reactions and in developing improved materials for bonding to niobium. This paper contains the results of the preliminary study carried out in this connexion.

Diffusion was studied (using the electron-probe microanalyzer to determine variations in composition across the diffusion zones) in the following couples: niobium/chromium; niobium/iron; niobium/nickel; niobium/molybdenum; niobium/18–8 (Type 304) stainless steel; niobium/18–12–Mo (Type 316) steel; niobium/18–10–Nb (Type 347) stainless steel.

Intermediate phases of stoichiometric composition were found in each of the binary couples. In niobium/chromium diffusion couples, the phases $NbCr_2$ and $NbCr_7$ were found to be always present, and, after long heating times, a NbCr phase with excess niobium in solid solution was also observed.

Study of niobium/iron diffusion showed the intermediate phase to be $NbFe_2$ plus excess iron in solid solution, but, depending on the impurity content of the iron, isolated niobium-rich compounds were determined far out into the iron.

Niobium/nickel couples often melted in the region of 1100°C., although reports in the literature give the eutectic temperature as 1175°C. Below 1095°C. the authors' observations revealed the presence of NbNi with excess niobium in solid solution, and Nb_2Ni precipitated in the NbNi matrix on cooling. Above 1095°C. (when the couple was on the verge of melting), rapid diffusion gave rise to zones up to 1400 microns in length, and $NbNi_3$ precipitated in a NbNi matrix on cooling.

Niobium and molybdenum were miscible at all compositions, and the diffusion coefficient ranged from 3 to 7 $\times$ 10^{-14} cm.^{2}/sec.

Diffusion of niobium with stainless steel resulted in the formation of a main zone (approximate composition: niobium 40, iron 40, chromium 8, nickel 5, wt. per cent.), which, it is speculated, contained a mixture of $NbFe_2$, $NbCr_2$ and NbNi. The data suggested that a small region near the niobium side of the diffusion zone contained a mixture of $NbFe_2$, NbCr and Nb_2Ni.

In the various systems studied, cracking often occurred in the diffusion zone, usually between the intermediate phases or near the niobium-poor side of the zone, but never near the niobium-rich side. The results indicate that temperature and the presence of impurities have a far greater influence on niobium binary diffusion than on niobium/stainless-steel diffusion. The molybdenum-containing steel exhibited less than the average degree of cracking.

Influence of Second-Phase Particles on the Corrosion of Nickel-containing Aluminium Alloys in High-Temperature Water

D. F. MACLENNAN: 'Corrosion of Aluminium Alloys in High-Purity Water in the Range 150°C.–340°C.'

Corrosion, 1961, vol. 17, May, pp. 239t–42t.

In previous work by the author, examination of corrosion-product surfaces formed after exposures of 1–5 minutes in water at 300°C. revealed differences in the corrosion pattern exhibited by commercial '2S' aluminium (99 per cent. aluminium) and experimental alloy '157'* (*ibid.*, 1961, vol. 17, Apr., pp. 181t–4t: see abstract in *Nickel Bulletin*, 1961, vol. 34, No. 6, p. 160). The results suggested a relationship between the corrosion patterns and the distributions of second-phase particles in the alloys. Additional information on the rôle of the second-phase particles, obtained by examining the corrosion product in cross-section while still attached to the metal, is presented and discussed in the present paper.

Specimens of the two alloys were exposed to high-purity water for 7 hours at temperatures ranging from 150° to 340°C. A coating of aluminium was then evaporated onto the outer surface of the corrosion product, to render it visible when photographed, and the specimens were mounted in plastic, cross-sectioned, polished, and then examined under an optical microscope.

The weight gain exhibited by the two alloys is plotted in the paper against the reciprocal of the absolute temperature, and photomicrographs are included to exemplify the condition of the specimens after corrosion.

The results obtained indicate that, in the early stages of corrosion, '2S' aluminium and the '157' alloy exhibited approximately the same corrosion rate, and that differences in their final behaviour were dependent

* Nominal composition: nickel 2, iron 0·5, silicon 0·2, per cent., remainder aluminium.

on the behaviour of the second-phase particles and the surrounding metal. The '2S' aluminium is shown to contain at least two types of second-phase particle, one of which, it is concluded, corrodes more rapidly, and the other more slowly, than the surrounding aluminium. In the case of the '157' alloy, the second-phase particles are far more numerous and a larger proportion corrode more slowly than the aluminium.

Corrosion-Resistance of Nickel-base Alloys in High-Temperature Nitrogen Environments

J. S. BRUNHOUSE and G. W. TITUS: 'Corrosion of Nickel-Based Alloys in High-Temperature Nitrogen Environments.'

Corrosion, 1961, vol. 17, May, pp. 203t-7t; disc., p. 207t.

Of 30 alloys evaluated in an exploratory survey of materials offering some possibility of proving suitable for use as cladding for the fuel elements of a mobile high-temperature gas-cooled reactor designed for the U.S. Army, the authors selected six solution- or precipitation-strengthened alloys for initial testing: the nickel-chromium-iron-base alloys 'Inconel' and 'Inconel X'; the nickel-chromium-base alloy 'Inconel 702'; the nickel-chromium-iron-molybdenum-base alloys 'Hastelloy X' and 'Hastelloy R-235'; and the nickel - molybdenum - chromium - iron - base alloy 'INOR 8' ('Hastelloy N'). The results of these screening tests are reported in the present paper.

The suitability of the alloys was assessed in relation to the degree to which they satisfied the main service requirements: satisfactory performance during extended operation (5000-10,000 hours) at a hot-spot temperature of 1750°F. (955°C.), in atmospheres either of air or of 99·5 vol. per cent. nitrogen + 0·5 vol. per cent. oxygen, under a maximum stress of 1000 p.s.i. Tubing not being readily available, the alloys were tested in the form of 0·05-in. (1·25-mm.) thick sheet. Short-time rupture tests were carried out at 1750°F. (955°C.), and elongation data were obtained in tests in which specimens were subjected to a stress of 2000 p.s.i. for 1000 hours at the same temperature. In corrosion tests, each alloy was heated for 1000 hours, under a stress of 1000 p.s.i., in a furnace pressurized to the operating pressure of the reactor (200 p.s.i.g.) by a slowly flowing air or coolant-gas atmosphere. Following exposure, specimens were weighed, sectioned and then examined microscopically, and some were subjected to tensile tests to obtain information on the effects of such exposure on strength and ductility. Micrographs illustrating microstructures before and after corrosion testing are included in the paper.

The data presented indicate that 'Hastelloy X' offers the most satisfactory combination of corrosion-resistance and strength under the test conditions, and this alloy has therefore been selected as the reference alloy toward which all design calculations and further evaluations will be directed. The performance of 'Inconel X' and 'Inconel 702' was deemed somewhat less attractive, but sufficiently promising for the alloys to be considered as alternative materials.

Corrosion of Stainless Steels in Boiling-Water Reactor Systems

See abstract on p. 188.

Properties of Boron-containing Chromium-Nickel Stainless Steels

K. BUNGARDT and R. OPPENHEIM: 'Properties of Boron-containing Chromium-Nickel Austenitic Stainless Steels for Service in Nuclear Reactors.'

Archiv f. d. Eisenhüttenwesen, 1961, vol. 32, Feb., pp. 95-101.

Boron-containing chromium-nickel stainless steels (chromium 18-20, nickel 11-15, per cent.) which, due to their boron content, possess a high thermal-neutron-absorption cross section are used for control rods and thermal shields in nuclear reactors. The investigation described in this paper (which opens with a review of the relevant literature) was undertaken to obtain further information on the influence of boron on the properties of chromium-nickel austenitic stainless steel.

The structure, hot workability and mechanical properties of a series of 18-11 chromium-nickel steels were investigated as a function of boron contents varying from 0·15 to 2·1 per cent. An 18-11-1·2 chromium-nickel-boron steel which finds application as a material for thermal shields was the subject of more comprehensive studies, which included evaluation of high-temperature properties and welding characteristics. Finally experiments were carried out to obtain data on the influence of chromium content on the corrosion behaviour of chromium-nickel-boron steels.

The results of the investigation indicate that boron-containing 18-11 steels are forgeable up to the eutectic boron content of about 2·1 per cent., but, in the case of larger ingots, the boron addition should be less than 1·5 per cent. Tensile strength and yield strength increase with increase in boron content, but elongation at rupture, reduction-in-area and notch impact strength are considerably reduced.

Steels containing chromium 18, nickel 11 and boron 1·2, per cent., were found to exhibit the following properties: tensile strength 65-80 kg./mm.^{2}, yield strength 40-50 kg./mm.^{2}, elongation 10-15 per cent., reduction-in-area 15-20 per cent., notch impact strength 2·5-4 kgm./cm.^{2}. Heat-resisting properties are good. Welding tests on plate up to 55 mm. thick confirmed that welds, especially those produced with boron-containing addition metal of similar type, possess satisfactory strength characteristics.

Since the numerous precipitated borides combine with considerable amounts of chromium, a study was made to determine whether it was necessary to modify the conventional composition of the steel and increase the chromium content as a function of boron content. In the case of chromium-nickel steels containing approximately 1·2 per cent. boron, the corrosion-resistance of a steel with 22 per cent. chromium and 10 per cent. nickel was found to be considerably higher than that of a steel with 18 per cent. chromium and 11 per cent. nickel.

Influence of Copper on the Corrosion Behaviour and Mechanical Properties of Chromium-Nickel Austenitic and Ferritic/Austenitic Steels

T. RAMCHANDRAN, K. ROESCH and H.-J. ENGELL: 'The Influence of Copper on the Corrosion Behaviour and Mechanical Properties of Chromium-Nickel Steels.' *Archiv f. d. Eisenhüttenwesen*, 1961, vol. 32, Mar., pp. 173-85.

The sparsity of data available on the influence of copper additions on the corrosion-resistance of high-alloy chromium-nickel steels prompted the authors to undertake a comprehensive investigation of the effects of up to 6 per cent. copper on the corrosion behaviour and mechanical properties of (1) austenitic steels nominally containing 18 per cent. chromium and 8 per cent. nickel, and (2) ferritic/austenitic steels containing 28 per cent. chromium and 4 per cent. nickel. Experiments carried out in an attempt to combine good corrosion-resistance with the improved mechanical properties obtained by precipitation hardening are deemed of particular interest.

The studies were conducted, and are reported, in two phases:

(1) The influence of copper additions on corrosion-resistance was evaluated by: (a) current-density/potential measurements at 80°C. in 2*N* sulphuric acid through which nitrogen was passed; (b) determination of loss-in-weight in various corrosive media; (c) study of susceptibility to intergranular corrosion using the boiling copper-sulphate/sulphuric-acid test.

(2) A study was made of the age-hardenability of steels of 28-4 chromium-nickel-copper type (18-8 steels could not be rendered age-hardenable), and the tensile, impact and creep properties of both types of steel were determined. Evaluations were also made of hot-working and welding characteristics.

The current-density/potential measurements and the weight-loss data demonstrated that, provided the copper remains in solution, the resistance of both types of steel to sulphuric acid increased with increase in copper content. Corrosion-resistance, however, decreased with increased precipitation of a copper-rich phase, which was preferentially attacked to a degree which increased as the temperature and acid concentration was raised. The passive dissolution current density attained a minimum value at copper contents in the range 3-4 per cent. The passivation current density fell steadily with increase in copper content, since precipitation of copper diminished the electrode surface and, hence, the current necessary for passivation. The 18-8 type austenitic steels exhibited an improved resistance to intergranular corrosion provided that the copper was in solution.

In the case of the 28-4 chromium-nickel steels, a definite age-hardening effect was observed at copper contents in the region of 4 per cent., and microscopical studies indicated that precipitation occurred only in the ferrite. Copper additions caused no age-hardening effects in steels of 18-8 type. While the tensile strength and elongation at rupture of the austenitic steels fell pronouncedly when free copper-rich phases were present in the structure, the tensile strength of the 28-4-type steels increased steadily with increase in copper content and elongation at rupture decreased. The notch impact strength of the austenitic steels was impaired by precipitation of a copper-rich phase. The creep behaviour of both types of steel was improved, within the limits of the short test times employed, with increase in copper content. The hot-working characteristics of both steels decreased with increase in copper content, though this effect was less pronounced with steels of 28-4 type. The 28-4 chromium-nickel steel exhibited good weldability at all the copper contents studied; the austenitic steel, on the other hand, exhibited a tendency to hot cracking at copper contents higher than 5 per cent.

Corrosion of Stainless Steels in Hot Hydrogen Sulphide

F. J. BRUNS: 'Welded Stainless Steels for Hot Hydrogen Sulphide Service.'

Corrosion, 1961, vol. 17, May, pp. 227t-31t.

This paper was originally presented at the 16th Annual Conference of the National Association of Corrosion Engineers, 1960, and was abstracted, from the preprint then available, in *Nickel Bulletin*, 1960, vol. 33, No. 7, pp. 178-9.

Corrosion Experience with Pulp-Mill Equipment

H. C. TEMPLETON: 'Corrosion of Stainless Steel and Higher Alloys in Pulp-Mill Equipment.'

Corrosion, 1961, vol. 17, June, pp. 9-13.

Pulp-making processes in established use include: (1) sulphite pulping systems (calcium-bisulphite, magnesium-bisulphite, sodium-bisulphite and ammonium-bisulphite liquors); (2) alkaline-soda and Kraft processes; (3) semi-chemical pulping (neutral sodium-sulphite, Kraft semi-chemical, cold-caustic and chemi-groundwood systems); and (4) mechanical or groundwood systems. Bleaching follows completion of the pulping process. In this paper the author presents (1) corrosion data obtained from test rods exposed to the liquors associated with these various processes, and (2) discusses corrosion experience with materials employed in pulp-mill equipment.

The corrosion data included in the paper relate mainly, though not exclusively, to the following: A.I.S.I. Type 304, 316 and 317 chromium-nickel stainless steels; 'Carpenter 20'; 'Monel'; nickel; 'Hastelloy B' and 'Hastelloy C'; 'Inconel'.

Corrosion of Stainless-Steel Pulp-Digester Linings

W. M. SCOTT: 'Corrosion of Stainless-Steel Pulp Digesters.'

Corrosion, 1961, vol. 17, June, pp. 14-16, 18.

In the sulphite process, digestion of wood is carried out at high temperatures in an aqueous solution which

usually contains an alkaline-earth bisulphite and an excess of sulphur dioxide. In a mill which uses limestone as its base, the cooking acid is calcium bisulphite. The acid is manufactured by melting and burning elemental sulphur to form the SO_2 gas. The gas is then cooled rapidly and passed upwards, through limestone towers, to unite with the calcium and form calcium bisulphite. Despite precautions, traces of sulphur trioxide are present in the cooled gas and in the raw acid. At some point in the process, some of the sulphur trioxide picks up another oxygen atom and unites with calcium to form calcium sulphate. In this paper the author discusses the pitting and stress-corrosion cracking believed to have been caused by calcium-sulphate scale deposited on the Type 316L (extra-low-carbon 18-12-2Mo) stainless-steel spot-welded lining of a sulphite digester at the British Columbia plant of Columbia Cellulose, Limited. Corrosion of the 'Hastelloy F' dome lining is also considered.

Theories are advanced in explanation of the corrosion damage, and repair procedures are described. Experience with a digester clad with Type 317 (18-13-3Mo) stainless steel is referred to, and freedom from trouble with this digester is attributed to improvements in plant operations and scale removing procedures.

It is recommended that stainless steel intended for sulphite service should contain a minimum of 2·75 per cent. molybdenum and a maximum of 0·05 per cent. carbon. All stainless-steel surfaces should be ground to a high finish and fully protected during manufacture.

Corrosion-Resistance of Stainless-Steel Overlays in Kraft Digesters

C. L. CARNS: 'Corrosion-Resistance of Stainless-Steel Overlays in Kraft Digesters.'

Corrosion, 1961, vol. 17, June, pp. 20–4.

In 1951, inspection of the bottom cone of one of the Kraft digesters at the Longview Mill (Washington) of the Weyerhaeuser Company revealed a localized reduction in shell thickness, and the region was overlaid, using Type 310 25-20 chromium-nickel stainless-steel weld metal. Since periodic inspection showed this repair, and subsequent repairs of a similar nature, to be in good condition, it was decided in 1956 to repair other units by overlaying with Type 310 steel applied by the automatic shielded-arc process. At the Company's Everett (Washington) plant, seven more digesters were overlaid in much the same manner, but, since failures had by then been noted in the upper parts of the units at the Longview Mill, Type 316 18-12-Mo stainless-steel weld metal was employed instead of Type 310. Analysis of these overlays revealed that the weld had been diluted by the basis metal in the ratio of 1 to 2. The efforts made to prevent such dilution, to find some means of decreasing the corrosion of overlays, and to develop repair procedures, form the subjects of the present paper.

Laboratory Corrosion Tests Simulating Service Conditions

A. O. FISHER: 'New Methods of Simulating Corrosive Plant Conditions in the Laboratory.'

Corrosion, 1961, vol. 17, May, pp. 215t-21t; disc., p. 221t.

Although information on the effects of temperature on the corrosion behaviour of materials of construction is of fundamental value to the designer of chemical processing equipment, relatively little published data are available. Iso-corrosion diagrams are usually based on generalized data, compiled from many sources and obtained under varied laboratory and field conditions, and the information is confined almost exclusively to the more common environments (such as sulphuric acid and nitric acid). Even more surprising than this lack of data is, in the author's opinion, the relatively few references in the literature to test apparatus suitable for use in obtaining a precise temperature/corrosion parameter for a given alloy in a specific environment. With the aim of indicating some of the most recent advances in such techniques, the author in this paper therefore gives details of laboratory corrosion-testing equipment developed for the express purpose of simulating service conditions as closely as possible. Particular emphasis is placed on corrosion under heat-transfer conditions, and the various devices are discussed in the light of the plant conditions which they were intended to simulate and the results which were obtained. The temperature/corrosion data presented are regarded merely as illustrative of the performance of the laboratory equipment.

Four devices are described: (1) A modified disc heat-flux apparatus suitable for reproducing corrosive plant conditions involving heat-transfer at temperatures up to 300°C.; (2) an elevated-temperature heat-flux corrosion apparatus; (3) a modification of the elevated-temperature heat-flux corrosion apparatus which permits evaluation of the effects of pressure up to 30 p.s.i.g. (135°C.); (4) an all-glass pressurized interface heat-transfer corrosion apparatus designed to permit simulation of the following exposure conditions: (a) heat-transfer surface (complete immersion); (b) heat-transfer surface (partial immersion); (c) cooling-condensing surface (partial immersion); (d) complete liquid immersion (control); and (e) complete vapour exposure (control).

Corrosion rates derived in investigation of specific plant problems (involving exposure to 93 per cent. sulphuric acid, boiling 70 per cent. nitric acid + 0·25 per cent. sulphuric acid, inhibited cooling water, and organic chemicals) are presented for mild steel, A-285 steel and various grades of chromium-nickel austenitic steel. The application of apparatus (4) is described, in some detail, in relation to the corrosion behaviour of the following materials in 65 per cent. nitric acid at high temperatures and pressures: Type 304, 309, 310 and 321 chromium-nickel stainless steels, 'Inconel', 'Multimet', 'Haynes 25', 'Hastelloy C', titanium and zirconium. Of these ten, zirconium alone offered promise of satisfactory performance under the plant conditions simulated.

Stress-Corrosion-Cracking Test for Copper-base Alloys

D. H. THOMPSON: 'A Simple Stress-Corrosion-Cracking Test for Copper Alloys.'

Materials Research and Standards, 1961, vol. 1, Feb., pp. 108–11.

Stress-corrosion cracking of copper-base alloys is believed to result from the simultaneous action of stress and a corrodent comprising ammonia, air and water. In this paper, the author gives details of an extremely simple stress-corrosion test for copper-base alloys, and presents test data in evidence of the 'quite reliable and informative results' which can be achieved by its means. On the credit side, the test is simple, requires very little equipment, and provides a quantitative measure of the progress of cracking; on the debit side are its inability to measure or vary the applied stress, and the use of cold-rolled specimens.

In the 'loop test', cold-rolled strip specimens of the alloy under study are bent around a mandrel until the ends touch. The ends are held together by a twist of copper wire (thus producing a tensile stress in the outer fibres at the bend), and the tear-drop-shaped loops are exposed to the vapours arising from concentrated ammonium-hydroxide solution at room temperature. Before and after exposure to the moist ammoniacal atmosphere, each loop is unfastened and allowed to spring out into its natural U-shape. The distance between the ends is measured, and the difference between the two measurements reflects the degree of stress-corrosion cracking. The author gives details of a standard test procedure, and data indicating the reproducibility of results are commented upon.

The test has been employed by the author to compare the resistance of 28 common copper-base alloys to stress-corrosion cracking in a moist ammoniacal atmosphere (using the time required to cause 50 per cent. relaxation as a criterion of resistance). The results tabulated indicate that alloys containing substantial amounts of zinc were most susceptible to cracking, followed by those containing aluminium. The highest resistance was offered by tough pitch copper and 70-30 cupro-nickel.

Selection of Materials for Use at Low Temperatures

A. HURLICH and J. F. WATSON: 'Selection of Metals for Use at Cryogenic Temperatures.'

Metal Progress, 1961, vol. 79, Apr., pp. 65–72.

The use of cryogenic fuels for missiles and space rockets has stimulated interest in the low-temperature properties of structural materials. Since the newer rockets under development in the U.S.A. will operate on liquid-oxygen and liquid-hydrogen fuels, work to overcome the problems associated with low-temperature applications can be expected to continue unabated. The authors, associated with Convair Astronautics Division of General Dynamics Corporation, California, were involved in such problems in connexion with the development of the Centaur space vehicle, the first missile in the U.S.A. to be fuelled with a liquid-oxygen/liquid-hydrogen combination. Designed as an upper-stage vehicle to be used with Atlas or Saturn boosters, the Centaur is intended for space exploration. Since the materials used in the vehicles are subjected to a variety of loading conditions at low temperatures during flight, accurate data on mechanical properties have to be obtained at temperatures down to —423°F. (—253°C.), the boiling point of liquid hydrogen. The cryogenic-test facilities established by the company for this purpose, and the results obtained on various sheet materials, form the subjects of this article.

The following three types of material are evaluated in relation to their suitability for low-temperature rocket applications: cold-rolled austenitic stainless steels; aluminium alloys; titanium alloys.

Machining of 18-8 Stainless Steel with Carbide Tools

H. TAKEYAMA: 'Performances of Carbide Tools in Machining 18-8 Stainless Steel.'

Amer. Soc. Mechanical Engineers, 1960, *Paper* 60-*W*-1; 7 pp.

The work reported, which formed part of a test programme designed to establish machining standards for various metals covered by Japanese specifications, was carried out to select the most suitable carbide grade for tools used in turning 18–8-type chromium-nickel stainless steel, to determine optimum tool shape, speeds and feeds, and to study tool life from the point of view of both crater and flank wear.

It is emphasized that, in determining machining conditions as a function of tool life, both crater and flank wear should be taken into account. In a light cut at higher speeds, tool failure is usually due to the crater, whereas, in a heavy cut at lower speeds, tool life is governed by flank wear. To establish optimum cutting conditions, it is therefore necessary to obtain tool-life curves for both crater and flank wear. These curves have been calculated, and in the present paper are used as a basis for determination of optimum speeds and feeds for turning 18–8 stainless steel.

Of the carbides studied, Grades MB2 (triple carbide No. 2) and MC1 (universal use No. 1) are considered most suitable for turning the steel (these designations are based on a new classification of carbide grades introduced by the author in an effort to standardize machining data). Under conditions of flank wear, use should be made of a side-rake angle of 0°–5°; if tool failure is governed by crater wear, the angle should be 10°–15°. For optimum tool life, feeds should not be less than 0·004 i.p.r.

The cutting fluid was found to have a marked influence upon tool life, and use of an appropriate fluid increased cutting speeds for 100-minute tool life (flank wear) by up to about 33 per cent. (optimum results were obtained with chlorinated oil or a chemical solution). The data indicate that most cutting

fluids have a more pronounced influence on flank-wear tool life than on crater. In turning 18–8 stainless steel, lubricity appeared to be a much more influential characteristic of the cutting fluid than cooling ability.

Wear of Carbide Tools in Machining 'Nimonic 80A'

W. S. McDOUGALL: 'Wear of Carbide Tools When Machining 'Nimonic 80A'.'

Department of Scientific and Industrial Research, Nat. Engineering Laboratory Plasticity Report No. 170, April 1960; 20 pp.

In an earlier report (*Report No.* 160, 1958) McDOUGALL and RAMSAY described experiments in which both the machinability and creep properties of two nominally identical bars of 'Nimonic 90' were found to differ significantly. Within the scope of a more general investigation of tool wear during machining of a range of materials under different cutting conditions, results have now been obtained on 'Nimonic 80A' which contribute to an understanding of the relatively high rates of tool wear encountered at relatively low cutting speeds.

Flank wear was studied in tests in which, using Grade T carbide tools (WC 74, Co 10, TiC 16 wt. per cent.), a solution-treated billet of 'Nimonic 80A' was cut orthogonally at speeds of 30 and 44 ft. per minute under dry conditions. Subsequently, studies were carried out [using tools made from Grade T and Grade E (WC 90, Co 10, wt. per cent.) carbides] to supplement the wear tests and provide information on the influence of cutting speed on chip formation, the values of components of cutting forces, and the built-up edge conditions. After the machining tests, the tools were subjected to metallographic examination.

The behaviour of 'Nimonic 80A' could, it is concluded, be related to the high values of cutting forces necessary to machine the material. The rapid increase in the proportions of total energy expended in friction at the flank face, at relatively small values of wear on flank face of the tool, emphasized the importance of this source of energy in the determination of temperature gradients. The work done against friction at the flank face was of the same magnitude as the work done against friction at the rake face and as the work of deformation at values of flank-face wear of 0·006 in. and 0·012 in., respectively. When the width of the wear band on the flank exceeded 0·012 in., serious chipping occurred at the cutting edge of the tool. Under the machining conditions used, the tool materials were deformed plastically at speeds in excess of 50 ft./min., the deformation being greater with tools made of Grade E carbide than with tools of Grade T carbide. The worn surface of the tool flank was covered with a film of workpiece material, and the nature of this film will require further investigation to assess its importance in relation to the other factors observed in wear studies of carbide tools when cutting 'Nimonic' and other materials.

Welding Metallurgy of the 'Nimonic' Alloys

J. HINDE and D. R. THORNEYCROFT: 'Welding Metallurgy of the 'Nimonic' Alloys.'*

HENRY WIGGIN & COMPANY, LTD., *Publication* 2209, 1961; 10 pp.

Reprint from *Brit. Welding Jnl.*, 1960, vol. 7, Oct., pp. 605–14.

See abstract in *Nickel Bulletin*, 1960, vol. 33, No. 11, p. 288.

Structure and Properties of Flash-Butt Welds in 'Nimonic' Alloys

F. A. BALL and D. R. THORNEYCROFT: 'The Structure and Properties of Flash-Butt Welds in 'Nimonic 75', '80A' and '90'.'

HENRY WIGGIN AND COMPANY, LTD., *Publication* 2160, 1961; 18 pp.

Reprint from *Welding and Metal Fabrication*, 1960, vol. 28, Sept., pp. 362–7.

See abstract in *Nickel Bulletin*, 1960, vol. 33, No. 12, p. 322.

Hot Ductility of 'Inconel' Weld Deposits Produced with 'BP-85' Electrode

W. A. OWCZARSKI: 'The Hot Ductility of 'Inconel BP-85' Weld Deposits.'

U.S. Atomic Energy Commission, Report KAPL-M-WAO-7, Apr. 1960; 20 pp.

The excellent resistance of 'Inconel' to stress-corrosion cracking has stimulated considerable interest in the advantages it offers in the field of nuclear power (e.g., as a material of construction for heat exchangers, primary-cooling-system piping, reactor-coolant pumps and valves, and for cladding the interior of the reactor vessel). It is essential in nuclear-power-reactor systems that weldments be free from defects, and the necessity of welding unusually heavy sections, sometimes several inches thick, has aggravated the welding problems involved.

In a previous report† the authors presented the results of a study of 'Inconel' weldments and overlays produced using commercially available 'Inconel'-type filler metals. All the filler metals studied were found to be capable of producing welds meeting the current naval nuclear standards, but all the welding processes and filler metals posed problems or difficulties which were regarded as limiting their application. The tendency to fissuring of the 'Inconel'-type filler metals was deemed worthy of further study. The International Nickel Company, Inc., has now developed a covered-electrode ('Inconel BP-85') which produces joints which bend tests have shown

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* *We shall be pleased to supply a free copy of this publication.*

† 'A Study of 'Inconel' Welding for Nuclear Power Service,' *U.S. Atomic Energy Commission Report KAPL-M-WAO-1*, July 1959; 54 pp.

to exhibit excellent resistance to fissuring. On the basis of their evaluation of the mechanical properties and corrosion-resistance of weld metal and weldments produced with the electrode (*Report KAPL-M-GFM-8*), the authors concluded that 'the BP-85' electrode is the best 'Inconel'-type covered electrode tested to date': the salient findings of this investigation were indicated in an abstract in *Nickel Bulletin*, 1960, vol. 33, No. 12, pp. 321-2. (A separate investigation of the degree to which the covered electrode, and a complementary inert-gas-shielded filler wire which deposits age-hardenable welds and overlays, satisfy nuclear-power requirements was reported by WITHERELL in *Welding Journal*, 1960, vol. 39, Nov., pp. 473s-8s; abstract in *Nickel Bulletin*, 1961, vol. 34, No. 2, pp. 45-6.) The work now described was undertaken to evaluate further the high-temperature properties of 'Inconel BP-85' weld deposits (in particular, tendency to fissure at high temperatures) on the basis of hot-ductility tests.

Eighteen longitudinal and six transverse specimens obtained from two butt joints produced between 'Inconel' plates were subjected to the R.P.I. hot-ductility test, and metallographic studies were also carried out. The weld metal composition is given as: nickel 65·42, chromium 15·05, iron 8·15, manganese 8·42, silicon 0·46, carbon 0·032, sulphur 0·006, titanium 0·60, niobium+tantalum 1·95, per cent.

On the basis of the data presented in the report, the authors conclude 'that 'Inconel BP-85' weld metal does have good hot ductility properties. This is borne out in other evidence which has been noted. Bend tests made from deposits in butt joints and overlays have generally been found to be fissure-free. Even though the tests are conducted at room temperature, the fissures which can be observed normally are formed during the deposition of a weld bead or by the shrinkage effects of subsequent beads. This would again be a 'hot' phenomenon, taking place at high temperatures. Liquid penetrant examinations of 'BP-85' welds or overlays have generally exhibited freedom from linear indications (of fissures). Weld-bead cracking or crater cracks have not been a problem when using the 'BP-85' electrode, even in areas of relatively high restraints.'

Advantages of Vacuum Melting in the Production of High-Temperature Brazing Alloys

C. R. BENSON: 'Vacuum Melting Produces Cleaner, Better-Performing High-Temperature Brazing Alloys.' *Welding Jnl.*, 1961, vol. 40, Mar., pp. 232-6.

While strict quality control over structural materials used in high-speed aircraft and missiles is established practice, virtually the only requirements that have been placed on brazing alloys used to join the assemblies are that they meet the specified composition and that they melt at the required temperature. Brazing alloys are usually manufactured in powder form, and quality depends to a great extent on the method by which they are produced. In this review the author discusses the advantages offered in this respect by vacuum-melting, and, in illustration of its effects, presents data on various vacuum-melted nickel-chromium-silicon-base high-temperature brazing alloys. Applications and the commercial availability of vacuum-melted brazing alloys are referred to.

Vacuum-melting is stated to produce 'cleaner' brazing-alloy powders essentially free from inclusions, undesirable impurities, surface oxides and absorbed gases. Erosion characteristics are improved and closer control over composition is achieved. The improvement in cleanliness ensures a more uniform and consistent performance in vacuum- or atmosphere brazing, and the lower gas content of vacuum-melted brazing powders is particularly advantageous in vacuum brazing.

ANALYSIS

Semi-micro Determination of Nickel with 4-Methylnioxime

See abstract on p. 178.

Spectrophotometric Determination of Nickel, Copper, Iron and Manganese

See abstract on p. 178.

Gravimetric Determination of Nickel Using Respropiophenone Oxime as a Reagent

See abstract on p. 178.

Determination of Nickel and Cobalt in Soils

See abstract on p. 178.

Spectrophotometric Determination of Major Amounts of Cobalt in Nickel Alloys

See abstract on p. 184.

Determination of Cobalt in High-Temperature Alloys

See abstract on p. 192.

Spectrophotometric Determination of Boron in Steel and High-Temperature Alloys

See abstract on p. 193.

Corrigenda

Nickel Bulletin, 1961, vol. 34, No. 3, p. 54.
'Low Temperature Mechanical Properties of Structural Materials: Compilation of Published Data', Line 1.
For H. P. GIBSON,
read H. P. GIBBONS.

Nickel Bulletin, 1961. vol. 34, No 5, p. 135.
'The Atmospheric Corrosion of Steels as Influenced by Changes in Chemical Composition', Line 23.
For Nickel: residual; 0·1
Read Nickel: residual; 1·0

Nickel Bulletin, 1961, vol. 34, No. 6, p. 149.

'Ferritic S.G. Iron Castings for Valves, Flanges, etc.', Line 4.

For A.S.T.M. Designation: A455-60T,
read A.S.T.M. Designation: A445-60T.

Ibid., p. 172.

'Aluminium-containing Free-Machining Stainless Steel', First Column of Table.

For Nitrogen
Read Nickel.

PATENTS

Extraction of Nickel, Cobalt and Iron from Laterite Ore

British Patent 855,544 claimed a process for the extraction of nickel and iron as carbonyls from a lateritic nickel-bearing ore by reducing the ore and then treating it with carbon monoxide. Any cobalt present in the ore remained in the solid residue from the treatment with carbon monoxide, and subsequent extractions by leaching with an ammoniacal solution of ammonium carbonate seldom exceed 80 per cent. According to the present patent, a lateritic nickel ore containing less than 10 wt. per cent. of magnesia is reduced by heating at an elevated temperature in a selectively reducing atmosphere so as to convert substantially all of the cobalt and the nickel contents of the ore, and a minor proportion of the iron, to metal. The reduced ore is cooled in a non-oxidizing atmosphere, and treated with carbon monoxide to volatilize most of the nickel and a small proportion of the iron as carbonyls. The residue is treated with dry chlorine gas to convert the remaining nickel and the cobalt to the water-soluble state, and the chloridized product is heated in an oxidizing gas containing free oxygen, to decompose any iron chlorides formed, and finally leached with water to remove substantially all the remaining nickel and cobalt.

INTERNATIONAL NICKEL COMPANY OF CANADA, LTD. *Brit. Pat.* 863,766.

Electrolytic Co-Precipitation of Metal Oxides

Metal oxides are co-precipitated by reacting the metal ions in a predetermined proportion with an alkali solution. The co-precipitation is carried out by electrolyzing (in the presence of oxygen or an oxidizing agent) an alkali-salt solution in which the anodes (fabricated from the metals which are to be precipitated as oxides) are separated from each other by insulating partitions (e.g., glass plates) which do not entirely extend up to the cathode. The anodes are used at a current strength proportional to the amount of the respective metal oxide required to be co-precipitated.

The process, which can be carried out continuously, is stated to offer advantages over chemical co-precipitation and mechanical mixing. The specification includes details of the procedure involved in the application of the invention to the production of a mixed oxide of NiO 84, MnO_2 16, per cent., suitable for use as colouring oxides in enamelling.

MAGNETO-CHEMIE N.V. *Brit. Pat.* 864,249.
Similar to German ,, 1,083,793.

Indirectly-Heated Nickel-Alloy Cathodes

An indirectly-heated cathode structure (such as a sleeve or cup), fabricated from an alloy of the following composition, is characterized by high strength at cathode-operating temperatures, sustained high level of electron emission, and low sublimation: zirconium 0·05–1·5 (preferably 0·1), tungsten 1–5 (preferably 4), aluminium 0–0·1, magnesium 0–0·07, wt. per cent., remainder nickel, and, optionally, cobalt 15 max., silicon 0·02 max., iron 0·1 max., manganese 0·1 max., manganese 0·1 max., carbon 0·08 max., copper 0·05 max., wt. per cent.

SUPERIOR TUBE COMPANY (inventors A. M. BOUNDS and R. L. HOFF). *Brit. Pat.* 866,194.
Similar to U.S. ,, 2,833,647.

Zirconium-Aluminium-Nickel Electron-Tube Cathodes

A cathode structure of the indirectly-heated non-filamentary type, including sleeves and cups, characterized by high-strength at cathode-operating temperatures, rapid activation, and good emission and sublimation characteristics, is composed of a nickel-base alloy free from tungsten and containing zirconium 0·05–0·5 wt. per cent. and aluminium 0·005–0·2 wt. per cent. Addition of zirconium and aluminium in the ranges claimed is stated to offer strength at operating temperatures substantially superior to, and emission and sublimation characteristics equivalent to, or better than, those of previously available nickel-alloy cathodes.

A preferred alloy contains: zirconium 0·05–0·5, aluminium 0·005–0·2, magnesium 0·07 max., silicon 0·05 max., carbon 0·08 max., manganese 0·15 max., copper 0·05 max., wt. per cent., remainder nickel.

A. M. BOUNDS and R. L. HOFF, assignors to SUPERIOR TUBE COMPANY. *U.S. Pat.* 2,975,050.

Nickel-containing Wear-Resistant Parts for Severe Service Conditions

The main aim of the invention is the provision of a wear part enabling a friction element to operate satisfactorily at high energy levels (as, for example, in intercontinental military aircraft, in which the friction element used for braking is suddenly subjected to severe torque and very high temperatures). At least the wear face of the part is composed

primarily of sintered powdered nickel aluminide or molybdenum aluminide bonded by a metal binder (preferably nickel). The part may also include a hard refractory material (nitrides, carbides, oxides or silicates, but, preferably, alumina).

AMERICAN BRAKE SHOE COMPANY. [*Brit. Pat.* 865,150.

Vitreous Enamel Free from Hydrogen Defects

A glass coating is applied to steel by a procedure involving: (1) blasting the steel with a metallic grit of 25–40 mesh, to provide an average surface roughness in the range 230–450 micro-inches; (2) depositing a layer of nickel on the roughened surface to a thickness of 0·08–0·3 gm./sq. ft.; (3) applying a coating of vitreous enamel directly to the nickel-plated steel surface; (4) firing the vitreous-enamel composition to fuse it to the steel.

The combination of grit blasting and nickel plating serves to eliminate fishscaling, and minimizes other hydrogen defects which normally occur in a glass coating applied to steel. In addition, the nickel coating ensures improved adherence between the glass and the steel, and obviates the necessity for incorporating adherence-promoting oxides in the glass composition.

J. D. SULLIVAN, assignor to
A. O. SMITH CORPORATION. *U.S. Pat.* 2,976,171

One-Coat Enamelling Process

The invention is based on the finding that a completely satisfactory one-coat-enamelled steel product is obtainable by subjecting it, prior to enamelling, to a treatment involving: (1) cleaning the surface with an alkali solution; (2) pickling (to remove 1·5–3 gm. of iron per sq. ft. of exposed surface) in a hot solution containing 10–30 per cent. phosphoric acid and having a controlled ferrous-ion content of 0·01–1 wt. per cent. and a ferric-ion content of 0·05–3; (3) washing the pickled surface with a weak alkaline solution (to remove the phosphate coating formed during pickling); (4) treating with a hot nickel-sulphate solution containing $NiSO_4 \cdot 7H_2O$ 1–4 oz./gal. and 0·001–0·03 wt. per cent. of phosphate ions, to deposit a nickel flash of 0·02–0·35 gm./sq. ft.; (5) rinsing the nickel-coated surface with an alkaline neutralizing or inhibiting solution, and then with clear water, before drying the surface in preparation for the application of a one-coat porcelain enamel.

E. M. OLIVER, H. P. TRIPP, L. E. FUSSELL and F. L. MICHAEL, assignors to GENERAL ELECTRIC COMPANY.
U.S. Pat. 2,977,241.

Nickel-containing Slip Composition for Protective Coatings

The paper covers a slip composition which, under standard temperature and pressure conditions, consists of: (1) about 70 parts by weight of a chromium-boron-nickel eutectic containing nickel 65–75, chromium 13–20, boron 3–5, iron, silicon and carbon 10 max., wt. per cent.; (2) about 30 parts of a ceramic frit consisting of BaO 44, SiO_2 37·5, B_2O_3 6·5, ZnO 5, CaO 3·5, ZrO_2 2·5, Al_2O_3 1, wt. per cent.; (3) about 5 parts clay (kaolin, enamelling clay or porcelain-enamel clay); (4) about 40 parts water.

Details are given of the procedures involved in applying the composition to the surface of a basis metal to provide, after firing, a protective coating stated to be highly resistant to corrosion, weathering, abrasion, thermal shock, etc.

D. G. MOORE, assignor to U.S. SECRETARY OF THE AIR FORCE. *U.S. Pat.* 2,974,051.

Protection of Combustion-Engine Exhaust Systems

The resistance of combustion-engine exhaust systems (e.g., car mufflers, tail pipes, etc.) to weathering and chemical attack is improved by chemically plating the interior and exterior surfaces of the hollow ferrous-base component with a coating of nickel, applying a coating of unfused porcelain enamel (containing an adherence-promoting oxide) to the exterior surface, and finally firing the component to form a fused porcelain-enamel layer on the exterior surface, and a uniform corrosion-resistant nickel-oxide-containing layer (the oxidation product of the electroless plating) on the interior surface.

E. E. BRYANT and C. J. REIFF, assignors to
FERRO CORPORATION. *U.S. Pat.* 2,975,072.

Deposition of Extremely Hard Nickel Coatings

The invention is based on the discovery that coatings produced by deposition of a metal upon a substrate comprising a binder (preferably polyisobutylene) and finely-divided grain-oriented graphite having a grain-size of the order of 0·1–25 micron are characterized by extremely high hardness (which is governed by the grain-size of the graphite and the degree of grain orientation). Electroformed nickel produced according to the invention (by a procedure described in the specification) is stated to be extremely hard-wearing and erosion-resistant (with a hardness ranging from 400 to 1000 Brinell) and to be suitable for use, for example, as a protective shield for the leading edge of an aircraft propeller blade.

METACHEMICAL PROCESSES, LTD.
(inventors, H. D. HUGHES and R. W. LEWIS).
Brit. Pat. 864,672.

Nickel Plating of Aluminium

The patent covers a method for plating an aluminium or aluminium-alloy substrate with a nickel coating, which then provides a suitable surface for subsequent deposition of an adherent coating of another metal. The advantages of the method include: the fact that only one underlying metal coating is required before depositing the desired finish metal; there are no rigid controls on dips and plating solution; excellent adhesion is attained; the finish metal can be deposited from a molten-salt bath.

The aluminium is degreased; rinsed in water; etched in hydrofluoric acid; rinsed in water; etched in a solution of hydrochloric acid containing manganous sulphate (the total etching time varying from 10 to 80 seconds); rinsed in water; nickel-plated in an aqueous solution containing nickel sulphate, magnesium sulphate, ammonium chloride and boric acid, at 15 amp./sq. ft. (1·62 amp./dm.²) for 10 minutes, to deposit a nickel layer 0·0001–0·0005 in. (0·0025–0·0125 mm.) thick; rinsed in water; partially dried with an air blast; heat-treated at about 1000°F. (540°C.) for 2½–5 minutes; anodically treated in a solution of sodium hydroxide and sodium carbonate to remove the light-blue oxide formed; rinsed in water; subjected to a reverse-current treatment in sulphuric acid; rinsed in water; and finally plated with a metal other than nickel (preferably gold).

J. C. WITHERS and P. E. RITT, assignors to MELPAR, INC. *U.S. Pat.* 2,970,090.

Chemical Nickel Plating of Silicate Glass

Uniform adherent films of nickel or cobalt (suitable, for example, for the production of soldered joints) are chemically deposited on silicate glass after a pre-treatment which involves chemically etching the glass, washing, activating the surface by alternately immersing it in an aqueous solution of a catalytic metal (nickel, cobalt or palladium) and in a solution of a reducing agent, and finally washing before plating. A submicroporous siliceous film, formed on the chemically etched surface of the glass by exposing it to HF vapour for 5–10 minutes, at a glass temperature sufficiently high to prevent condensation of moisture, provides an effective anchorage for subsequent deposition of nickel or cobalt from a solution containing a nickel or cobalt salt, hypophosphite ions and a carboxylic-acid salt.

J. MOCHEL, assignor to CORNING GLASS WORKS. *U.S. Pat.* 2,968,578

Method of Improving the Corrosion-Resistance of Chemically-Nickel-Plated Magnesium-base Alloys

According to the claims of the patent, the corrosion-resistance of nickel coatings chemically deposited (from an aqueous solution containing nickel and hypophosphite ions at pH 3·5–7) onto a magnesium-base alloy is improved by immersing the plated alloy in an aqueous solution (pH 0·1–8; temperature 60°–212°F.) containing a chromium compound selected from chromic acid (0·1–5 wt. per cent.), soluble dichromates and soluble chromates (5–15 wt. per cent.), removing the alloy and washing with water.

H. K. DE LONG and D. E. RITZEMA, assignors to DOW CHEMICAL COMPANY. *U.S. Pat.* 2,975,073.

Cobalt-Nickel-Chromium Alloy for Use in Point Bearings

Point bearings (e.g., for watches) stated to be of better and more reproducible quality than those produced from martensitically hardened material are made from wire in an alloy of the following composition: cobalt 40–50, chromium 10–20, molybdenum about 5, tungsten about 5, beryllium 0·2–0·4, titanium 0·8–1·2, manganese + silicon about 2, iron 0–10, per cent., remainder nickel. The wire is homogenized at 1100°C., quenched to atmospheric temperature (the unhardened condition), cold drawn to a strength of at least 200 kg./mm.² and a hardness of 600 Vickers, age-hardened at 400°–500°C., and finally cut into short lengths in the form of bearings having pointed ends.

R. STRAUMANN, assignor to INSTITUT DR. ING. REINHARD STRAUMANN A.G. *U.S. Pat.* 2,973,291.

Manganese-Copper-Nickel-base Corrosion-Resistant Alloys with High Coefficient of Expansion and High Electrical Resistance

The alloys covered by the patent were developed to provide materials which, while exhibiting the advantages of those containing manganese 72, copper 18, nickel 10, per cent. (which are widely used because of their high coefficient of expansion and high electrical resistance), offer a lower temperature coefficient of resistance and improved corrosion-resistance.

The alloys claimed consist of (1) about 72 per cent. manganese, 14 per cent. nickel, 9 per cent. chromium and 5 per cent. copper (the alloy being heated for several hours at 750°C., to reduce the temperature coefficient of resistance to about 0·00002 in the range 20°–150°C.); (2) about 16·5–18 per cent. copper, 9·5–10·5 per cent. nickel, 0·5–3 per cent. aluminium, and about 72 per cent. manganese, the alloy exhibiting a coefficient of linear expansion at 20°C. of about 30×10^{-6}/°C.

R. S. DEAN, assignor to CHICAGO DEVELOPMENT CORPORATION. *U.S. Pat.* 2,982,646

Hard Surfacing Titanium with Titanium-Nickel-Copper Alloys

An erosion-resistant coating is applied to the surface of a component (preferably a titanium-base steam-turbine blade) subjected to erosion or abrasion during service by depositing (preferably by an inert-gas-shielded arc-welding process) a eutectiferous alloy consisting of titanium and (1) nickel 20–35 and copper 0–10, per cent., or (2) manganese 35–45 per cent., or (3) cobalt 20–32 per cent., or (4) silicon 6–10 per cent.

IMPERIAL CHEMICAL INDUSTRIES, LTD. (inventors, D. C. MOORE and E. A. TAYLOR). *Brit. Pat.* 869,197.

Titanium-Nickel-Copper Welding or Brazing Rods

Welding or brazing rods of eutectic or eutectiferous titanium-base alloy (preferably containing nickel 20–35, copper up to 10, per cent.) are produced by mixing the alloying constituents in powder form (at a

particle size $<250\mu$), compacting into sheet or plate having a density at or approaching the theoretical density, sintering the compact in an inert atmosphere or *in vacuo* at a temperature (preferably 650°–800°C.) compatible with the attainment of adequate machinability, and finally machining the sheet or plate into rods.

Rods prepared as described were melted satisfactorily by inert-gas arc-welding techniques and are suitable for use in hard-facing titanium.

IMPERIAL CHEMICAL INDUSTRIES, LTD. (inventor, A. E. SALT). *Brit. Pat.* 869,198.
Similar to Canad. „ 601,712.

Glass-to-Metal-Sealing Alloys

The alloys, which lie along curves extending into the homogeneous γ solid-solution region of the iron-nickel-cobalt ternary system, are stated to exhibit no tendency to martensite formation (with its adverse effects on electrical and expansion characteristics) during processing or service.

In the case of alloys for soft-glass/metal seals, the composition curve passes through three points, corresponding to: (1) iron 39·5, cobalt 34·5, nickel 26, per cent.; (2) iron 41, cobalt 18, nickel 41, per cent.; (3) iron 44·5, cobalt 4, nickel 51·5, per cent.

For hard-glass/metal seals, the three points of the curve correspond to: (1) iron 54, cobalt 17, nickel 29, per cent.; (2) iron 55·5, cobalt 10, nickel 34·5, per cent.; (3) iron 57·5, cobalt 2·5, nickel 40, per cent.

H. WLOKKA. *East German Pat.* 18,461.

Iron-Nickel-Manganese Temperature-Responsive Compensator for Magnetic Instruments

The patent covers a temperature-responsive magnetic alloy which, containing nickel 29·25–31·25, manganese 1–2·4, silicon $<0·5$, chromium $<0·3$, per cent., remainder iron, has a magnetic permeability which decreases with increase in temperature. The small amount of manganese present improves low-temperature stability by shifting the gamma/alpha phase boundary and increasing the proportion of the alloy in the gamma phase.

The alloy is intended for use, as a single-compensator material, in correcting errors due to temperature changes in magnetic measuring instruments (e.g., speedometers).

R. H. MITCHELL and R. E. SCHWYN, assignors to GENERAL MOTORS CORPORATION. *U.S. Pat.* 2,975,339.

Low-Alloy Steels Free from Alligatoring

Steels containing about 1·5–2·5 per cent. manganese, 0·75–3 per cent. chromium and 0·75–3·5 per cent. nickel exhibit high impact- and wear-resistance, but are subject to 'alligatoring' when cast in sections larger than about 4 in. (10 cm.). The patent claims a high-impact and wear-resistant low-alloy steel for heavy-section castings which is rendered free from alligatoring by inclusion of minimal amounts of silicon and rare-earth metals. Silicon is present in a minimum amount of 0·6 per cent., and the steel is de-oxidized by the addition to the molten metal of at least about 0·1 per cent. aluminium, and at least about 0·35 per cent. rare earth, based on the weight of molten metal.

C. G. MICKELSON and C. D. ASHERMAN, assignors to AMERICAN STEEL FOUNDRIES. *U.S. Pat.* 2,970,903.

Ductile High-Strength Low-Alloy Steel

A high-strength low-alloy steel exhibiting a high strength/weight ratio, and with isothermal-transformation characteristics adequate to permit it to be easily worked in its metastable austenitic condition without causing strain-induced transformation during working (and thus to resist cracking during forming or upon cooling), comprises carbon 0·45–0·55, total manganese, molybdenum and vanadium 0·5–2, silicon 1·75–2·25, nickel 4·5–7·5, chromium 0·75–1·25, per cent., remainder essentially iron.

W. H. CHANG, assignor to GENERAL ELECTRIC COMPANY. *U.S. Pat.* 2,978,319.

Welding Wire for High-Strength Low-Alloy Steels

A welding wire of the following composition is intended for use in gas-shielded electric-arc welding low-alloy steels of high yield strength and impact-resistance: carbon 0·13 max., silicon 0·3 – 0·5, phosphorus, 0·025 max. sulphur 0·025 max., manganese 1·7–2·3, nickel 0·4–0·8, molybdenum 0·25–0·5, per cent., remainder iron. The weld is stress-relief annealed at 1200°F. (650°C.) for 1 hour.

The wire's primary advantage is that it produces a weld deposit which, after heat-treatment (such as stress-relief annealing), exhibits yield-strength and impact-resistance values matching those of the low-alloy-steel basis metal. Welds in 'HY-80' steel are stated to exhibit a yield strength of 94,000 p.s.i. (42 t.s.i.: 66 kg./mm.^{2}) after stress-relief treatment for 1 hour at 1200°F. (650°C.), a value similar to that for weld metal in the as-welded condition; the impact strength of the weld is considerably improved by stress-relief treatment and values of 55 and 90 ft. lb. were obtained at —60° and +20°F. (—50° and —7°C.) respectively.

A. MULLER and C. R. FELMLEY, assignors to AIR REDUCTION COMPANY, INC. *Canad. Pat.* 613,358.

Welding of 9 per cent. Nickel Steel Without the Need for Stress Relieving

In welding 9 per cent. nickel steel plate, it has been found that *A.S.T.M.* side-bend specimens cut from weldments produced with the joint contours hitherto used cannot be successfully bent in the as-welded non-stress-relieved condition. Attempts to obviate the trouble and cost involved in stress-relief treatment (which, in the case of 9 per cent. nickel steel welded

pressure vessels, is mandatory under present Code requirements*) have resulted in the development of a weld-joint construction which permits the production of weldments satisfying side-bend-test requirements in the as-cast condition and which obviates the need for pre-heating.

The invention involves the following stages: (1) the abutting faces of the two 9 per cent. nickel steel plates to be welded are shaped to an in inclination of 7°-12° with respect to the centre line of the weld joint; (2) each of the root faces extending from the inclined surfaces to the bottoms of the plates is formed on radii of approximately $\frac{5}{16}$ in. (8 mm.) relative to the centre line of the weld joint; (3) a predominantly nickel root-weld metal is deposited in beads so as to extend upwardly $\frac{5}{32}$-$\frac{3}{16}$ in. (4-5 mm.) from the bottom of the plates in the root, the root side of the weld-joint width being substantially equal to the narrowest width of the joint on the face or initial side of the weld joint; (4) the space between the inclined sides is filled by depositing beads of predominantly nickel welding material between them.

The steel plates are of the following composition: carbon 0·13 max., manganese 0·8 max., phosphorus 0·035 max., sulphur 0·04 max., silicon 0·15-0·3 (ladle analysis), 0·13-0·32 (check analysis), nickel 8·5-9·5 (ladle analysis), 8·4-9·6 (check analysis), per cent.

The composition of the root weld and filling weld is selected from the following: (1) carbon 0·15 max., manganese 1-3·5, iron 6-12, sulphur 0·02 max., silicon 0·75 max., copper 0·5 max., chromium 13-17, niobium 1-3, molybdenum 0·5-2, per cent., remainder nickel+cobalt; (2) carbon 0·1 max., manganese 2-2·75, iron 10 max., sulphur 0·015 max., silicon 0·35 max., copper 0·5 max., titanium 2·5-3·5, chromium 14-17, cobalt 1 max., nickel 67 min., per cent.

W. L. NEELY, assignor to LUKENS STEEL COMPANY.
U.S. Pat. 2,982,018.

Purification of Nickel- or Nickel-Cobalt-base Heat-Resisting Alloys

Nickel-base or nickel-cobalt-base creep-resisting alloys, containing chromium, and rendered precipitation-hardenable by aluminium and titanium additions, are purified by locally superheating the surface of the molten alloy (to a temperature above the boiling point of the impurities) by contact with the plasma of an electric arc in an inert-gas atmosphere (preferably argon, and preferably in a stream which, directed towards the metal surface, protects it from oxidation and carries away the volatilized impurities) at atmospheric pressure. The bath is preferably stirred by electric induction, and where impurities are present which are more easily removed by oxidation than volatilization, part of the surface of the bath may be exposed to an oxidizing atmosphere to which heated metal is caused to flow from the heated part of the bath.

The creep properties of such alloys are impaired by trace amounts of lead, and the invention is stated to be particularly applicable to minimizing the amount of lead present (e.g., from an initial content of 8 p.p.m. to a final one of less than 2 p.p.m.).

MOND NICKEL COMPANY, LTD.* (inventor, J. H. GITTUS).
Brit. Pat. 866,078.

Nickel-base Heat-Resisting Alloy for Precision Castings

The patent relates to alloys which, castable into components suitable, in the as-cast condition, for use at temperatures up to 1900°F. (1035°C.), and preferably melted *in vacuo*, contain cobalt 9-11, chromium 9-15, tungsten 6-10, titanium 3·5-5, aluminium 3-5, carbon 0·04-0·2, boron 0·02-0·2, zirconium 0·01-0·2, wt. per cent., remainder (except for impurities, which should not exceed 3 per cent.) nickel. Iron may be present in amounts up to 2 per cent., manganese up to 0·5 per cent., and silicon up to 0·5 per cent. Oxygen and nitrogen should not exceed 0·01 per cent. each.

The alloys (1) are castable, by precision-casting techniques, into parts conforming to close tolerances; (2) are resistant to oxidation in air at temperatures up to 1800°F. (980°C.); (3) are capable of sustaining stresses of over 15,000 p.s.i. (6·7 t.s.i.; 10·5 kg./mm.^{2}) at temperatures up to 1800°F. (980°C.) for at least 100 hours (with an elongation of at least 3 per cent.); (4) exhibit good room-temperature tensile strength, with substantial ductility; and (5) have a high modulus of elasticity over a range of temperature.

WESTINGHOUSE ELECTRIC CORPORATION.

	Brit. Pat.	863,912.
Similar to U.S.	,,	2,951,757.
,, ,, *French*	,,	1,221,245.
,, ,, *Belgian*	,,	576,392.

Nickel-base High-Temperature Casting Alloy

A nickel-base alloy, suitable for casting, and exhibiting high stress-rupture strength and good tensile ductility (particularly in the range 2000°-2150°F. (1095°-1175°C.)) contains: chromium 14-16, aluminium 4·3-5·0, titanium 3·25-3·75, molybdenum 3·5-4·0, tungsten 1·4-4·1, boron 0·05-0·1, iron 6 max., zirconium 0·1 max., carbon 0·08 max., per cent., remainder nickel.

Data indicate that alloys falling within the scope of the invention have exhibited a stress-rupture life of 35 hours under a stress of 3,300 p.s.i. at 2130°F. (1165°C.), and a tensile reduction-in-area at 1300°F. (705°C.) of 17-19 per cent.

J. E. WILSON and E. L. DUNN, assignors to GENERAL ELECTRIC COMPANY. *U.S. Pat.* 2,975,051.

* In this connexion attention may be drawn to papers reporting the results of tests on pressure vessels fabricated from 9 per cent. nickel steel (see abstracts and table in *Nickel Bulletin*, 1961, vol. 34, No. 3, pp. 66-7). The tests demonstrate that 9 per cent. nickel steel exhibits, in the quenched-and-tempered condition, low-temperature notch toughness equal or superior to that of the double-normalized-and-tempered material called for in present Code requirements. The steel was shown to be notch-tough with either heat-treatment, and to resist brittle fracture even in the presence of the pronounced residual stresses encountered in as-welded vessels, a finding which should, it is felt, make it possible to omit the stress-relieving treatment now required after welding.

* Now renamed The International Nickel Company (Mond), Limited.

Nickel-base Age-Hardenable Alloy Suitable for Working from Large Ingots

A high-strength heat-resisting alloy of the following composition is dually hardened by hot working (the alloy may be hot worked into massive shapes) at 2000°-2300°F. (1095°-1260°C.) to produce a finely dispersed boride in the matrix, solution-treating at 1850°-2250°F. (1010°-1230°C.) long enough to produce a matrix of a finely dispersed second phase, cooling to room temperature, ageing for 5-200 hours at 1200°-1700°F. (650°-925°C.), and cooling in air to room temperature: chromium 12-28, molybdenum 7-25, aluminium 1-6, boron 0·2-0·8, vanadium 0·2-1, manganese 0·2-2, carbon 0·08 max., iron up to 20, wt. per cent., remainder nickel.

The hardened alloy exhibits high strength and ductility in the range 1300°-1500°F. (705°-815°C.): e.g., at 1400°F. (760°C.) a stress-rupture life of 8 and 227 hours, and elongations of 21 and 41 per cent., at a stress of, respectively, 65,000 (29 t.s.i.: 45·5 kg./mm.^{2}) and 35,000 p.s.i. (15·5 t.s.i.: 24·5 kg./mm.^{2}).

J. T. BROWN, assignor to WESTINGHOUSE ELECTRIC CORPORATION. *Canad. Pat.* 618,610.

Forgeable Heat-Resisting Austenitic Iron-base Alloys

The alloys covered by the patent are stated to offer the following advantages (and, on that account, to be suitable for use, for example, as wheels in gas or steam turbines): (1) high strength at temperatures in the region of 1200°F. (650°C.), and even as high as 1500°F. (815°C.); (2) ready forgeability at temperatures in the range 1950°-2100°F. (1065°-1150°C.); (3) corrosion-resistance at temperatures in the range 1200°-1500°F. (650°-815°C.); (4) satisfactory resistance to thermal shock.

The alloys contain: chromium 10-20, nickel 18-50, tungsten 1·5-8, niobium and/or tantalum 1-5, titanium 2-4, carbon 0·05-0·25, per cent., remainder essentially iron. The alloys may also contain cobalt up to 10, boron up to 0·1, zirconium up to 0·3, per cent.

SIERRA METALS CORPORATION. *Brit. Pat.* 866,839.
Similar to Canad. ,, 616,871.

Heat-Hardenable Fully Austenitic Stainless Steel

The aim of the invention is to provide 'a heat-hardenable, fully austenitic stainless steel, employing only a minimum of strategically important components, and, as well, to provide a method of heat-hardening the same which itself is simple and direct, which steel is clean, is readily worked or formed in the pre-hardened condition, which is weldable with ease, and which displays high strength properties at both room and elevated temperatures'.

The following composition range is claimed: chromium 10-30, nickel 0·01-18, manganese 0·01-16 (nickel+manganese 6-30), vanadium 0·5-2, carbon 0·2-1·5, molybdenum up to 4, tungsten up to 4, niobium up to 1·5, copper up to 4, nitrogen up to 0·75, boron up to 0·005, per cent., remainder iron. The carbon serves as an austenite former in the pre-hardened condition, and the vanadium and carbon combine, upon heat-treatment, to form a hardening carbide throughout the austenite matrix.

H. TANCZYN, assignor to ARMCO STEEL CORPORATION. *Canad. Pat.* 614,514.

Chromium-Manganese-Nickel Heat-Resisting Stainless Steel

A principal aim of the invention is the provision of a steel of low strategic alloy content, which can be readily forged into such heavy parts as jet-engine discs, and which can be precipitation-heat-treated (at temperatures which are not objectionably high) to give high strength at elevated temperatures (e.g., at 1200°F. (650°C.) and higher), with uniformity of properties throughout the full cross-section.

The steel contains carbon 0·1-0·4, nitrogen up to 0·3, phosphorus 0·15-0·35, chromium 9-12, manganese 4-20, nickel 4-10, per cent., remainder iron. The carbon+nitrogen content should be at least 0·15 per cent., and sulphur may be present up to 0·15, tungsten and molybdenum up to 3·5, boron up to 0·005 and vanadium up to 0·75, per cent. The following preferred composition is claimed: carbon 0·1-0·4, phosphorus 0·15-0·35, chromium 12, manganese 8, nickel 8, molybdenum up to 3·5, nitrogen up to 0·3 (carbon+nitrogen at least 0·15), per cent., remainder iron.

W. C. CLARKE, assignor to ARMCO STEEL CORPORATION. *Canad. Pat.* 614,929.

Chromium-Nickel Precipitation-Hardenable Stainless Steel

Stainless steels of the following composition, which are austenitic (and thus easily fabricated) upon annealing in the range 1850°-2000°F. (1010°-1095°C.), are strengthened by transformation to martensite (e.g., by refrigeration), and ageing at 800°-1100°F. (425°-595°C.): carbon 0·06-0·2, manganese 0·5-5, silicon 1 max., chromium 12-20, nickel 1-7, molybdenum 4 max., tungsten 4 max., vanadium 1 max., phosphorus 0·4 max., copper 0·5-3, boron 0·01-0·7, per cent., remainder iron. The nickel plus manganese content should not exceed 7·5 per cent. An advantage of the steel is stated to be the use of boron, copper and phosphorus as precipitation-hardening additions in place of such highly oxidizable elements as aluminium or titanium.

Among the data presented in illustration of the steel's characteristics are the following indicating properties in the hardened condition: tensile strength, 205,000 p.s.i. (91·5 t.s.i.: 144 kg./mm.^{2}); 0·2 per cent. offset yield strength, 185,000 p.s.i. (82·5 t.s.i.: 130 kg./mm.^{2}); elongation in 2 in., 7 per cent.

A. M. HALL, D. C. LUDWIGSON, D. B. ROACH and V. W. WHITMER, assignors to REPUBLIC STEEL CORPORATION. *U.S. Pat.* 2,967,770.

Nickel-containing Cermet Resistant to Ultra-High Temperatures

Cermets covered by the patent are capable of withstanding ultra-high temperatures for short periods of time, and are suitable for use in such applications as nose cones for guided missiles, throats for rocket motors, and supports for high-temperature filaments in inert gases, vacuum tubes and mercury rectifier tubes. A feature of the cermet is its ability to vaporize at high temperatures without passing through the liquid phase, and to form a vapour blanket around the component, so shielding it from the surrounding high temperatures.

The cermet consists of a blend of discrete particles of magnesium oxide, tungsten and nickel in compacted form, the particles being surface bonded to form a rigid mass, in which, however, their identity is maintained. The constituents are present in about the following percentages: magnesium oxide 22, tungsten 70, nickel 8, per cent.

E. WASHKEN, L. A. RUNTON and H. C. MORTON, assignors to RUSSELL MANUFACTURING COMPANY.
U.S. Pat. 2,952,903.

Coating Nickel-base-alloy Turbine Blades with a Protective Layer of Aluminium Alloy

In coating nickel-base- or cobalt-base-alloy turbine blades with an oxidation-resistant aluminium-nickel or aluminium-cobalt layer by aluminium dipping and subsequent diffusion treatment, some excess aluminium gives rise to the formation of a thin aluminium and aluminium-oxide overlayer which has to be removed by etching, a procedure which involves risk of attack on the aluminium-nickel layer. The present invention provides an acid pickling solution which, while effectively removing the aluminium overlay, is suitably buffered or inhibited to decrease its rate of attack on the protective alloy-layer.

After application of a thin aluminium or aluminium-alloy layer, the turbine blade is subjected to a diffusion heat-treatment, and the solidified coating is then treated in an aqueous pickling solution containing 7–11 vol. per cent. of hydrochloric acid and 70–100 g./L. of trisodium phosphate for 1–2½ hours to remove substantially all the coating which has not been diffused. The blade is then heated to diffuse the coating metal further into the basis metal.

J. L. GREENE and J. C. HOLZWARTH, assignors to GENERAL MOTORS CORPORATION. *U.S. Pat.* 2,970,065.

Electrodeposition of Aluminium on Nickel-base Alloys

A thin ductile oxidation-resisting aluminium-alloy coating is formed on a nickel- or cobalt-base alloy by: heating the basis alloy at 1250°–1600°F. (675°–870°C.); immersing it in a fused-salt bath at 1250°–1600°F. (the fused salt bath is in contact with a molten aluminium anode and comprises approximately 0·5–12 wt. per cent. aluminium fluoride, 8–20 wt. per cent. cryolite, 25–45 wt. per cent. potassium chloride and 37–57 wt. per cent. sodium chloride); imposing a negative potential on the alloy so as to induce a current density of about 0·1–30 amp./sq. in. and to electrodeposit aluminium onto the surface at a rate not substantially greater than that at which aluminium diffuses into the article; and removing any residual salts from the surface.

The invention is stated to be particularly suitable for coating the surface of high-temperature components (e.g., turbine blades) with an oxidation-resistant layer.

D. K. HANINK and A. A. SHOUDY, assignors to GENERAL MOTORS CORPORATION. *U.S. Pat.* 2,971,899.

Brazing of Nickel-base High-Temperature Alloys

The aim of the invention covered is the provision of a relatively simple and non-critical fluxless method of brazing nickel-base alloys (containing between about 3 and 8 per cent. total of aluminium and titanium) without the necessity of nickel-plating the surfaces to be brazed.

The components to be brazed are first heat-treated to oxidize preferentially the aluminium and titanium in their surface zones. (The components are preferably heated at approximately the solution-heat-treating temperature of the alloy, and, in the case of 'Inconel 702', 'Inconel X', 'Inconel 700', 'Nimonic 80', 'Nimonic 80A'. 'Nimonic 90', 'Nimonic 95' and 'Hastelloy R-235', a suitable temperature range is given as approximately 1900°–2200°F. [1035°–1205°C.].) The oxide scale formed during heat-treatment is then removed from the surface zones (preferably by pickling in a solution containing water 1000, nitric acid 1000, hydrofluoric acid 150, cc.), leaving the remainder substantially intact. During the oxidizing treatment a surface layer 0·0005–0·002 in. thick is depleted of aluminium and titanium, and removal of the oxide scale then exposes this depleted layer, which presents a surface forming oxides readily dissolved in dry argon or helium atmospheres at brazing temperatures. After the oxide scale has been removed, the components are therefore brazed in an inert atmosphere consisting of argon or helium having a dew point at least as low as −100°F. (−73°C.) (and preferably between −160° and −180°F.: −107° and −118°C.).

R. E. PRITCHARD and H. W. HOEFER, assignors to ORENDA ENGINES LIMITED. *U.S. Pat.* 2,957,239.

Aluminium-Nickel-Iron Alloys Resistant to High-Temperature Water

Aluminium-base alloys containing iron and/or nickel are produced by compacting the alloy granules (with a total iron and/or nickel content of 0·05–4 per cent.), and extruding to produce a shearing effect in at least the portion adjacent to the surface, and to ensure an even distribution and fine dispersion of the alloying metal in the aluminium. Procedures for producing the alloy granules are claimed. The sheared portion of the component exhibits improved resistance to high-temperature water.

Alloys covered by the patent are stated to resist water at 350°C. and 160 kg./cm.² for 5000 hours with no deterioration, to be resistant to cold water, and to exhibit no tendency to localized corrosion.

COMMISSARIAT A L'ENERGIE ATOMIQUE.
Brit. Pat. 868,769.

Iron-Nickel-Silicon Hot-Workable Acid-Resistant Alloy

The patent claims an iron-nickel-silicon alloy containing nickel 28–60 (preferably 35), silicon 9–12·2 (preferably 10), wt. per cent., remainder iron, and a process for the production of articles in the alloy, which comprises casting an ingot of the required composition, heating it to the temperature range 900°–1075°C., and hot working it in that range to the desired shape.

The invention is stated to satisfy the demand for a high-silicon alloy combining good resistance to corrosion by acids (e.g., sulphuric acid) with satisfactory hot workability.

UNION CARBIDE CORPORATION (assignee of R. A. PERKINS and C. R. BISHOP).
Brit. Pat. 868,112.
Similar to French „ 1,244,661.

Nickel-Molybdenum-base Acid-Resistant Alloys of Improved Ductility

In nickel-molybdenum and nickel-molybdenum-chromium heat- and corrosion-resisting alloys, the presence of the NiMo intermetallic as a second phase is beneficial to high-temperature strength, but (being of a hardness about twice that of the nickel-molybdenum solid-solution series) deleterious to ductility. According to the present patent, alloys of the following composition (preferably produced by melting at a pressure of 0·1 Torr and a temperature of 1650°C. for at least 30 minutes) have a tensile strength comparable to that of known nickel–molybdenum–base alloys, and, due to the absence of the NiMo phase, a considerably higher ductility: molybdenum 19–32, (or molybdenum 10–19 and chromium 10–18), carbon 0·01–0·03, silicon 0·05–0·6, iron 0·1–2, per cent., remainder nickel.

O. JUNKER and K. SCHERZER.
British Pat. 869,753.
Similar to Austrian „ 209,974.
„ „ *French* „ 1,184,118.

Nickel-Iron-Chromium Alloy Resistant to Reducing Acids

The alloys covered are stated to exhibit resistance to reducing acids (such as hydrochloric acid and sulphuric acid) approaching, or equalling, that of materials of higher alloy content, and ductility comparable to that of corrosion-resistant steels of lower alloy content. They therefore offer the advantages of high corrosion-resistance and satisfactory ductility at relatively moderate cost. Data exemplifying corrosion-resistance are included in the specification.

The alloys, which are readily weldable, contain: nickel 35–45, chromium 10–15, molybdenum 4–10, copper 3–5, silicon <3, manganese <2·5, carbon <0·1, per cent., remainder essentially iron. The following preferred composition is claimed: nickel 40·06, chromium 13·18, molybdenum 6·05, copper 3·98, silicon 0·73, carbon 0·04, manganese <2, per cent., remainder essentially iron.

N. S. MOTT (assignor to COOPER ALLOY CORPORATION). *Canad. Pat.* 619,135.

Aluminium- and Sulphur-containing Free-Machining Stainless Steel*

The invention is based on the finding that a free-machining austenitic steel can, by addition of sulphur and aluminium, be produced without the deleterious effects on hot-working characteristics associated with inclusion of sulphur alone. The patent covers an austenitic chromium-nickel steel containing chromium 15–30, nickel 6–40, carbon up to 0·25, sulphur 0·05–0·2, aluminium 0·25–1·35, wt. per cent.

UNIVERSAL-CYCLOPS STEEL CORPORATION.
Brit. Pat. 869,690.
Similar to U.S. „ 2,900,250.

Stainless-Steel Radiators for Domestic Heating Systems

In a domestic hot-water heating system of 'direct' type (in which fresh water is continuously being added to the system to replace that drawn off at the taps), corrosion of the pipes and radiators, and discoloration of bath water, are minimized by fabricating the tubing from copper and the radiators from chromium-nickel austenitic-steel sheet (chromium > 10 per cent.; nickel > 5 per cent.). To prevent galvanic corrosion, the radiators are fabricated by welding and manufacturing processes which ensure that the similarity between the electrolytic potential of the stainless steel and copper is maintained.

H. G. BINDER. *Brit. Pat.* 855,232.

* The reader's attention is drawn to an article, abstracted in Nickel Bulletin, 1961, vol. 34, No. 6, p. 172, which gives details of the properties of, and experience with, 'Uniloy 303MA' steel which falls within the scope of the patent.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Trade Marks*.

A Publication prepared by the
DEVELOPMENT AND RESEARCH DEPARTMENT
of
THE INTERNATIONAL NICKEL COMPANY (MOND) LIMITED

PRINTED IN ENGLAND *by* WIGHTMAN MOUNTAIN LTD.
WESTMINSTER, LONDON, S.W.1